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ERDEC-TR-415

CLEANOUT AND DECONTAMINATION OF A MUSTARD AGENT TON CONTAINER

Darren W. Dalton

ENGINEERING DIRECTORATE

June 1997

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The U.S. Army's Chemical Stockpile Disposal Program established the Alternative Technology Program to develop an alternative (chemical neutralization methods) to destroy the bulk blister agent HD (mustard) due to public concerns about the possible effects of incinerator emissions. ERDEC and the office of the Product Manager for Alternative Technologies and Approaches is working to develop the data required to assess whether an alternative technology should be tested in a pilot-scale facility. One of the critical aspects of the program is the successful decontamination of ton containers (TCs), which are used for bulk storage of the agent. During initial experiments for the incineration process at Johnston Atoll, solid residue ("heel") was observed in the HD TCs after draining the liquid agent. The HD heel presented a major problem in decontaminating TCs with an alternative technology. It was critical to decontaminate the TCs to a safe level of vapor agent concentration (3X condition) so that the TCs could be shipped to Rock Island Arsenal, treated to ensure a 5X condition, and smelted. The U.S. Army's alternative technology process focused on decontaminating HD TCs using pressurized hot water and steam. ERDEC has successfully decontaminated two HD TCs in an ERDEC Toxic Test Chamber to a 3X condition using this process.

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FOREWORD

This demonstration report provides detailed information regarding the cleanout and decontamination of a second mustard agent (HD) ton container (TC) conducted in a Toxic Test Chamber (TTC) in Building E3566 at the Edgewood Research, Development, and Engineering Center (ERDEC) in Aberdeen Proving Ground (APG) - Edgewood Area (EA). This demonstration focused on the cleanout steps after the liquid HD agent was drained from a TC. Its main objectives were to dislodge and remove residues left inside the TC and to decontaminate the interior of the TC to a 3X condition (vapor space HD concentration less than 0.003 mg/m^3) by flushing the eductor tubes with steam and spraying the interior of the TC with pressurized hot water. The demonstration was designed to confirm the results of the first HD TC Cleanout Demonstration, and to determine the minimum amount of water and time required to remove the "heel" and to reach a 3X condition. The demonstration was also designed to determine if the effluent can be recycled through the high pressure system once the "heel" was removed.

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PREFACE

The work described in this report was authorized under Chemical Agent Munitions Destruction Defense (CAMDD). This work was started in July 1996 and completed in November 1996.

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Department of the Army

U.S. Army Program Manager for Chemical Demilitarization
Alternative Technology Program

Ton Container Decontamination and Disposal Program Demonstration Report
A Second Mustard Agent Ton Container (TC Serial #D94102)

27 November 1996

Product Manager for Alternative Technologies
and Approaches

Date

Test Director, Product Manager for Alternative Technologies
and Approaches

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Date

Team Leader, Edgewood Research Development,
and Engineering Center

2 Dec 96
Date

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CLEANOUT AND DECONTAMINATION OF A MUSTARD AGENT TON CONTAINER

SECTION 1 EXECUTIVE SUMMARY

1. EXECUTIVE SUMMARY

1.1 Introduction

In response to public concerns about the possible effects of incinerator emissions on the local environment and health, the U.S. Army's Chemical Stockpile Disposal Program (CSDP) established the Alternative Technology Program (ATP) to develop an alternative to incineration for destroying two chemical warfare agent [blister agent HD (mustard) and nerve agent VX]. This project was later expanded to evaluate three vendor-developed alternative technologies. Any alternative destruction process selected must, at a minimum, meet the requirements of safety, environmental protection, and cost effectiveness set forth by Congress [Public Law (PL) 102-484]. The research and development (R&D) of an alternative technology is being pursued for possible use at bulk-only storage sites where TCs filled with either HD or VX are stored. At these sites, explosives are not a factor. These chemical agent stockpiles are, according to CSDP's current schedule, to be destroyed by incineration by the end of 2004.

One of the objectives of the ATP is to research low-temperature, low-pressure chemical agent destruction processes based on chemical neutralization and biodegradation of the reaction products. The office of the Product Manager for Alternative Technologies and Approaches (PMAT&A) is working to develop the data required to assess whether an alternative technology should be tested in a pilot-scale facility. An Overarching Integrated Product Team (OIPT) is scheduled to make a recommendation in December 1996 to proceed with the pilot program.

HD and VX are stored in bulk (ton) containers at two storage sites, HD at Aberdeen Proving Ground (APG) - Edgewood Area (EA), Maryland, and VX at Newport Chemical Depot (NECD), Indiana. A major concern associated with the ATP neutralization process is the agent residue remaining inside the TC after draining of the bulk liquid agent. The residue that cannot be pumped or drained out of the TC is called a "heel". The Ton Container Decontamination and Disposal Program Demonstration Plan: Mustard Agent Ton Container (Reference 3) detailed procedures to demonstrate that pressurized hot water could dislodge and remove residues inside an HD ton container (TC) and decontaminate the TC to a 3X condition, which is defined as a vapor space HD concentration less than 0.003 mg/m^3. The first demonstration was conducted in the

Toxic Test Chamber (TTC) in building E3566 at the U.S. Army's Edgewood Research, Development, and Engineering Center (ERDEC) in Edgewood, Maryland during January and February 1996. This demonstration was designed to provide information for the design of the chemical agent demilitarization pilot plant. A second demonstration, conducted in July 1996, was necessary to confirm the results of the first demonstration and to provide additional information for the design of the chemical agent demilitarization pilot plant.

1.2 Objectives

The Test and Evaluation Master Plan (TEMP) for the CSDP ATP (Reference 6) lists TC decontamination as one of the critical technical parameters of the program.

The main objective of this second demonstration was to confirm the concept that a high pressure hot water spray could remove residual solids and liquid from inside an HD TC and render it to a 3X condition. Also, additional information necessary for the design of the chemical demilitarization pilot plant was needed, and included the minimum amount of water and time required to remove the solids and achieve a 3X condition, and assessing the feasibility of recycling the effluent once the solid heel was removed. As part of the cleaning operations, initial solid residues, liquid effluents, undissolved solid materials, and vapors were sampled, quantified, and characterized.

1.3 Demonstration Summary

The demonstration system consisted of a high pressure hot water generator that could spray up to 5 gallons per minute (gpm) of 194°F (90°C) hot water at a maximum pressure of 3000 pounds per square inch (psi). The hot water discharge line was connected to a spray lance and nozzle assembly mounted along the centerline of the TC. Effluents were continually pumped out of the TC during the cleaning operations through a drain line inserted into the TC. The pump removed any water or residue that could have impeded cleaning. The effluents were strained with a coarse (1/4 inch perforations) duplex strainer ahead of the pump and a fine (80 mesh) duplex strainer after the pump. Also, after the solid heel was removed, a settle tank with a weir and an additional pump were installed ahead of the effluent pump. Once strained, the liquid effluent was transferred to a 316 stainless steel (SS) holding tank.

The "worst case" TC (Serial #D93734) was used for the first HD TC Cleanout Demonstration since it was identified by non-destructive evaluation (NDE) as having the largest amount of heel (13.5 inches). The HD TC selected for this demonstration (Serial #D94102) was identified by NDE as having an 11 inch heel. This TC was drained of liquid agent.

As part of this demonstration, neat agent was analyzed for purity, landban compounds and flashpoint. Characterization samples were analyzed for HD agent concentration as well as the concentration of metals and complex organic compounds. The cleanout effluents were analyzed for HD agent concentration, principal reaction products [thiodiglycol (TDG) and other products], metals, and other compounds. Vapor samples were analyzed for HD agent concentration and volatile organic compounds (VOC's).

1.4 Demonstration Results

A ten inch hole was cut in one end of the TC to allow access to the TC interior and for cleaning the TC interior. Before cleaning the interior of the HD TC, a vapor sample of the TC head space was taken and the interior of the TC was observed. The TC vapor primarily consisted of HD and 1,2-dichloroethane. The HD heel, which was determined to be approximately 359 pounds, was similar in appearance to the heel that was observed in the first HD TC; however, much more heel was present in this TC than in the "worst case" TC identified by NDE. A visual inspection showed that the heel was approximately 8 to 9-1/2 inches thick and was relatively uniform along the bottom of the TC. The heel consisted of extremely hard brown and black colored clumps or chunks similar to stalagmites. No observable liquid agent remained in the TC. A distinctive line, rust-to-tar, which indicated the previous liquid HD agent level, was also observed.

Each eductor tube was flushed with steam prior to cleaning the interior of the TC. The TC's interior was then sprayed with high pressure (approximately 2600 psi) hot water (approximately 195°F) at a flow rate of 5 gallons per minute (gpm). Samples of liquid and solid, if present, effluents were collected at planned intervals, which were dependent on the type of sample and the stage of cleaning. Although equipment problems were encountered, the demonstration basically proceeded as planned. No solids were collected in the strainers after the 10 minutes of hot water spraying. The level of vapor contamination inside TC was determined at 30 minute intervals using Depot Area Air Monitoring System (DAAMS) tubes. After 30 minutes of hot water spraying (152 gallons of water), the solid heel was no longer present and the interior of the TC appeared visibly clean; however, the vapor level of HD agent concentration indicated that the TC was not in a 3X condition. The concentration of HD agent in the liquid effluents decreased to below 200 parts per billion (ppb) after 45 minutes of hot water spraying. It took a total of 10 minutes of steam flushing the eductor tubes and 60 minutes of hot water spraying the interior of the TC to achieve a 3X condition. A total of 315 gallons of water was needed to clean and decontaminate the inside of the HD TC. The entire TC was then monitored for HD agent to ensure that the entire TC was in a 3X condition as well.

The initial solid heel was comprised of mainly iron salts, HD, and a solid cyclic sulfonium ion, which confirmed the results of the first HD TC cleanout. Once

pressurized hot water was sprayed into the TC, the iron salts and the cyclic sulfonium ion dissolved, and the HD hydrolyzed to TDG.

The liquid effluents were primarily composed of the cyclic sulfonium ion, TDG, and 1,4-dithiane. The initial pH of the liquid effluent was below 1; however, the pH increased to between 2 and 3 after the heel was removed after 30 minutes of hot water spraying. After the second 30 minutes of hot water spraying, the pH detected was between 5 and 6.

The sample from the settle tank (which was installed after the first 30 minutes of hot water spraying) was analyzed for particle size distribution. Results indicated that approximately 57 weight (wt) percent of the particles collected ranged from 0.007 to 0.02 inches, 10 wt% ranged from 0.006 to 0.007 inches, and 23 wt% were smaller than 0.006 inches. The remaining 10 wt% of the particles were greater than 0.02 inches. The particles resembled iron oxide flakes. Results from a total suspended solids analysis indicated that there were 270 to 280 parts per million (ppm) of suspended solids in the effluent stream after 60 minutes of hot water spraying.

1.5 Conclusions and Recommendations

The cleanout and decontamination of an HD TC containing approximately 359 pounds of residue were successfully demonstrated using steam and pressurized hot water, which confirmed the results of the first demonstration. A total of 10 minutes of flushing the eductor tubes with steam and 30 minutes of hot water spraying the interior of the TC (162 gallons of condensate and water) was necessary to remove the heel. A total of 10 minutes of flushing the eductor tubes with steam and 60 minutes of hot water spraying the interior of the TC (315 gallons of water) was necessary to achieve a 3X condition. No results were obtained that would indicate that this process is incompatible with the operation of an HD neutralization plant. It is therefore recommended that flushing the eductor tubes with steam and then spraying the interior of the TC with pressurized hot water be used in the pilot plant design to cleanout and decontaminate HD TCs.

Data were also obtained to assess the feasibility of recycling the effluent once the solid heel was removed. Results indicated that this effluent can be recycled.

If the effluent is recycled in the pilot plant, it is recommended that appropriate strainers be placed in line to protect the pumps and to keep the spray nozzle from clogging. Also, it has been observed that it is critical to spray closer to the plug and eductor tube walls to thoroughly clean the TC ends.

SECTION 2
BACKGROUND

2. BACKGROUND

2.1 Introduction

Due to public concerns about the possible effects of incinerator emissions on the local environment and on health, the U.S. Army's Chemical Stockpile Disposal Program (CSDP) established the Alternative Technology Program (ATP) to develop an alternative to incineration for destroying stockpiled bulk chemical warfare agents. The United States' stockpile of chemical warfare agents, which includes blister agent HD (mustard) and nerve agent VX, is contained in a variety of munitions and bulk containers. These chemical warfare agents are distributed among eight stockpile sites in the Continental United States and Johnston Atoll in the Pacific Ocean.

One of the objectives of the ATP is to research low-temperature, low-pressure chemical agent destruction processes based on chemical neutralization and biodegradation of the reaction products. This project was later expanded to evaluate three vendor developed alternative technologies. Any alternative destruction process selected must, at a minimum, meet the requirements of safety, environmental protection, and cost effectiveness set forth by Congress [Public Law (PL) 102-484]. The research and development (R&D) of an alternative technology is being pursued for possible use at bulk-only storage sites where TCs filled with either HD or VX are stored. At these sites, explosives are not a factor. These chemical agent stockpiles are, according to CSDP's current schedule, to be destroyed by incineration by the end of 2004.

The office of the Program Manager for Alternative Technologies and Approaches (PMAT&A) is working with the Edgewood Research, Development and Engineering Center (ERDEC) in conducting this R&D program. An Overarching Integrated Product Team (OIPT) is scheduled to review the results of the R&D effort in December 1996 to determine whether to pursue pilot plant operations of an alternative technology. The review must be completed by that time to prevent delay in the CSDP schedule for destruction of the stockpiled agents at APG-EA and NECD.

A major concern associated with the ATP neutralization process is the agent residue remaining inside the TC after draining of the bulk liquid agent. The residue that cannot be pumped or drained out of a container is called a "heel". In the baseline process (incineration), the agent residues are thermally converted to their combustion products when the TC is treated in the Metal Parts Furnace (MPF). This leaves the empty TC in an agent-free 5X condition, suitable for transport, direct recycling as scrap metal, and uncontrolled public access. A 5X condition is achieved when an item is maintained at

1000°F (537°C) for at least 15 minutes. For the chemical neutralization process, an incineration step for TC decontamination is not available.

The major concern for TC cleanout and decontamination by a nonthermal method is the unknown character of agent, particularly HD, subjected to a long term storage in uncontrolled environments. Corrosion and chemical degradation processes have resulted, leaving behind accumulations of solid residues, some of which may be absorbed or chemically bonded into the interior surfaces or surface coatings of the TC.

In preparation for the first demonstration, background information was gathered in the Ton Container Decontamination and Disposal Program Special Studies Report (Reference 1). This report includes results of a literature search on TC contents and a review of existing and potential methods for TC cleanout. The methods for each of the process steps were presented in the Ton Container Decontamination and Disposal Program Process Selection: Engineering Evaluation/Best Technical Approach Report (Reference 2) and evaluated against a defined set of selection criteria. This report concluded that pressurized hot water followed by steam will be the most effective method for cleaning and decontaminating HD TCs. The Ton Container Decontamination and Disposal Program Demonstration Plan: Mustard Agent HD Ton Container (Reference 3) detailed procedures to demonstrate pressurized hot water (followed by steam, if necessary) could dislodge and remove residues inside an HD TC and decontaminate the TC to a 3X condition. For HD, a 3X condition is defined by Army Regulation (AR) 385-61 as vapor space HD concentration less than 0.003 mg/m^3 when measured in a closed container (space) with an Automatic Continuous Air Monitoring System (ACAMS®), Miniature Continuous Air Monitoring System (MINICAMS®), or Depot Area Air Monitoring System (DAAMS). The HD vapor space concentration of 0.003 mg/m^3 is also defined as 1 time-weighted-average (TWA).

In January and February 1996, the first HD TC Cleanout Demonstration was performed on the "worst case" TC (serial #D93734.) This TC was selected using non-destructive evaluation techniques. TC #D93734 had the largest amount of heel of the 300 HD TCs evaluated. The results of the first demonstration were documented in the Ton Container Decontamination and Disposal Program Demonstration Report: Mustard Agent Ton Container (Reference 7) which indicated that pressurized hot water and steam was capable of removing the solid residue and decontaminating the interior of the TC to a 3X condition. Also, it was concluded that the eductor tubes need to be flushed in order to achieve a 3X condition. Since optimal conditions (mainly inlet water flow rate and temperature) were not present throughout the first demonstration, it was recommended that a second HD TC Cleanout Demonstration be performed to confirm the results of the first demonstration and to obtain additional information that was needed for the pilot plant design.

2.2 Objectives and Criteria

The Test and Evaluation Master Plan (TEMP) for the CSDP ATP (Reference 6) lists the TC decontamination as one of the critical technical parameters of the program. The main objectives of this demonstration were to confirm the results of the first HD TC Cleanout Demonstration and to provide additional information for the pilot plant design. More specifically, it was necessary to confirm the effectiveness of flushing the eductors with steam to remove solids and residue inside the eductor tubes, and the effectiveness of pressurized hot water spray to remove the heel/residue inside the TC and decontaminate the TC to a 3X condition. The composition of the TC heel also required confirmation. In addition, there was a requirement to determine the minimum amount of water and time required to (1) remove the heel and (2) decontaminate the TC to a 3X condition, and to assess the feasibility of using recycled water through the high pressure system after the heel was removed.

The overall HD TC cleanout process, as conceived for the pilot plant, includes HD thawing, venting pressurized vapors, draining liquid agents for processing, rinsing with a hot water spray to remove the heel, cutting the TC, decontaminating the TC to a 3X condition with pressurized hot water and/or steam, and preparing the decontaminated TC for shipment for final disposal. The unproven steps in this overall process were the use of a pressurized hot water spray to remove agent residues from the TC and pressurized hot water and/or steam to decontaminate the TC. The first demonstration proved that a combination of steam and pressurized hot water was capable of removing agent residues from the TC and decontaminating the TC.

2.3 Summary of Differences between Demonstrations

The first and second HD TC cleanout demonstrations used the basic approach and philosophy. The differences between the original Ton Container Decontamination and Disposal Program Demonstration Plan: Mustard Agent Ton Container (Reference 3) and the plan for cleaning a second HD TC were documented on a Demonstration Change Form, dated 3 July 1996, and approved by PMAT&A on 8 July 1996 (Appendix C).

As stated above, determining the minimum amount of time and water required to remove the HD heel and achieve a 3X condition, and assessing the feasibility of recycling water were new demonstration objectives.

The equipment modifications included using an 80 mesh duplex strainer instead of a 40 mesh duplex strainer for the fine strainer. The 80 mesh strainer had openings with diameters of 0.007 inches, which corresponds to the diameter of the spray nozzles. Also, a settle tank and an additional pump were installed after the solid heel was removed. The 80 mesh duplex strainer and the settle tank were used to assist in

assessing the feasibility of recycling the effluent. The TC was vented during the eductor tube flushing and the ten minute soak period during the pressurized hot water spraying.

Procedural changes included first, flushing each eductor tube separately with steam and then, hot water spraying the interior of the TC. Also, for each pressurized hot water spraying cycle (30 minutes), the nozzle was to be located at the three-quarter position for 15 minutes and at the one-quarter position for the remaining 15 minutes.

The number of samples collected for analysis was optimized. This resulted in a reduced number of samples. The methanol impinger samples were no longer required since it was determined that the vapor sample collected in the SUMMA® canister would be sufficient. Other analytical changes included neat HD agent being analyzed for purity, metals, organic compounds, and flash point; and the settle tank sample being analyzed for particle size distribution. It was also decided that volatile organic compounds from the DAAMS samples were no longer needed since the vapor samples taken in the SUMMA® canisters would provide the information required.

SECTION 3
SUBTEST #3
MUSTARD AGENT TON CONTAINER (Serial #D94102)

3. SUBTEST #3: MUSTARD AGENT TON CONTAINER (Serial #D94102)

This section provides detailed information regarding the setup, procedures, and the results obtained from a second HD TC Cleanout Demonstration, in which HD TC Serial #D94102 was cleaned and decontaminated using only steam and pressurized hot water.

3.1 Demonstration Objectives

The following were specific objectives for the second HD Ton Container Cleanout Demonstration:

a. Confirm ton container heel composition and mass by quantifying and characterizing the residue remaining in the HD TC immediately following TC draining.

b. Confirm the effectiveness of flushing the eductor tubes with steam, prior to hot water spraying the TC interior, to remove solids and residues.

c. Confirm the effectiveness of a high (up to 3000 psi) pressure impingement water spray using hot (90±5°C) water to remove solids and scale from the inside walls of an HD TC to achieve visibly clean surfaces.

d. Determine the minimum quantity of water and time required to remove the heel for a visibly clean TC.

e. Determine the minimum quantity of water and time required to clean the TC to a 3X condition (HD vapor concentration less than 0.003 mg/m^3).

f. Assess the feasibility of using recycled water through the high pressure system after the TC is visibly clean by measuring:
   - particle size distribution >.007" in the recycle stream
   - fluid pH in the recycle stream
   - fluid organic composition
   - particle composition

g. Quantify and characterize all solid, liquid, and vapor effluents from pressurized hot water cleaning.

3.2 Criteria

This demonstration was to be considered successful if the entire interior surface of the TC was visibly clean, the level of HD agent vapor concentration was below 1 TWA (0.003 mg/m^3), and the following were obtained:

a. Plots or profiles of all raw data versus time - temperatures, pressures, flow rates.

b. Level of contamination after each stage of pressurized hot water cleaning and at the end of the demonstration.

c. Samples of solids prior to cleaning operations. Components will be identified and quantified.

d. Samples of liquid effluents and solid materials during cleaning operations. Components will be identified and quantified.

e. Samples of vapors before and during cleaning operations. Components will be identified and quantified.

f. Completed data collection sheets.

3.3 Demonstration Description and Setup

The demonstration was performed using an HD TC that was stored and drained at the Chemical Transfer Facility (CTF). The HD TC (Serial #D94102) was selected because its' heel size was similar to the first HD TC and it was more cost and time effective to use this TC instead of moving a TC from the Chemical Agent Storage Yard (CASY). 300 CASY TCs were evaluated (heel size and fill level) by nondestructive techniques in May 1995, while the CTF TC was evaluated in July 1996. TC #D94102 had a heel size of 11 inches. The "worst case" heel was measured at 13.5 inches.

The demonstration system, as shown in the schematic in Figure 3-1 and the photograph in Figure 3-2, was developed for the HD TC Cleanout Demonstration, and was assembled from commercially available components. Operators inside the E3566 Toxic Test Chamber (TTC), dressed in appropriate personal protective equipment (PPE), performed the demonstration by operating the system and collecting the required samples.

HD TON CONTAINER CLEANOUT DEMONSTRATION

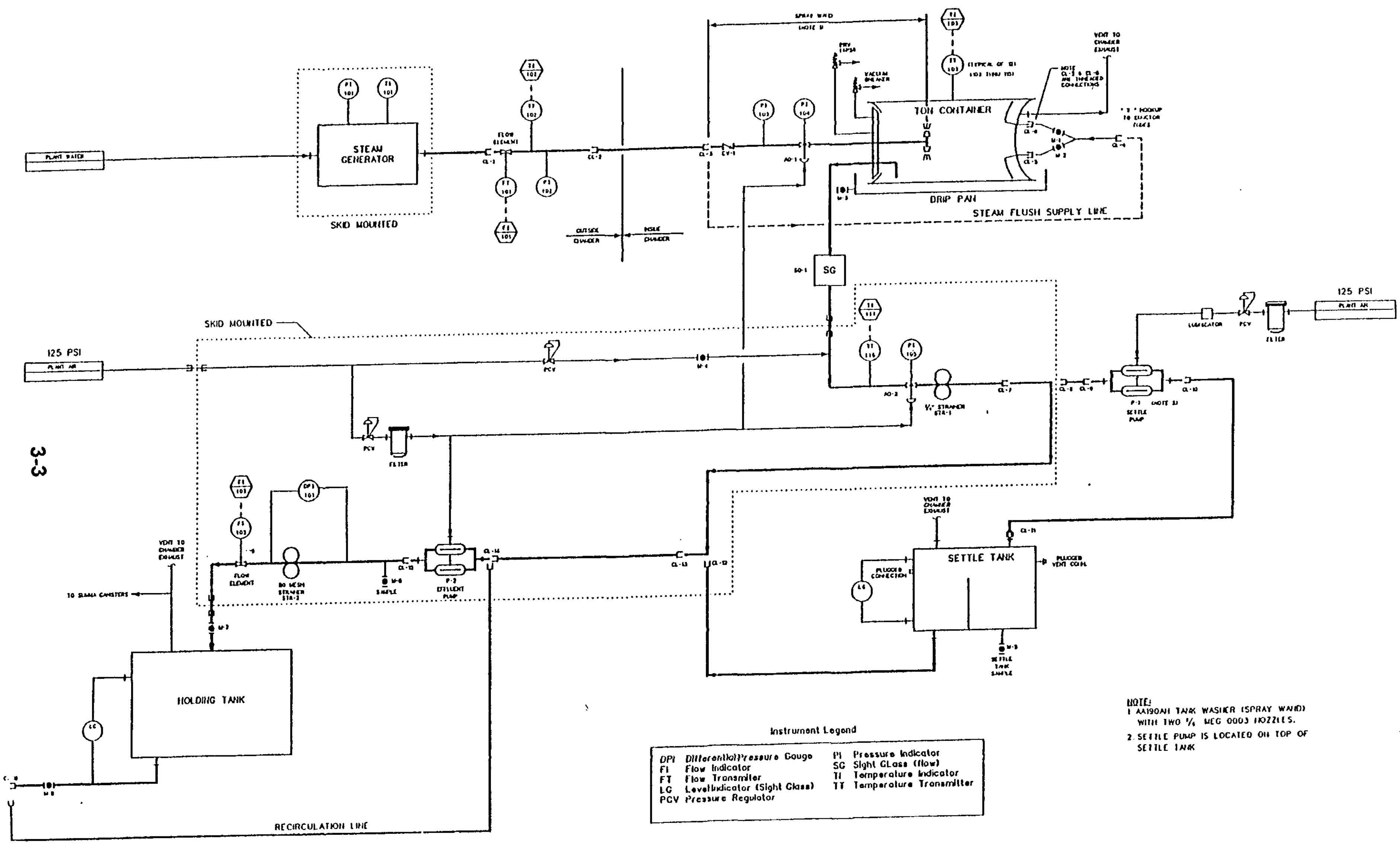


Figure 3-1. As Built Drawing for the Second HD TC Cleanout Demonstration

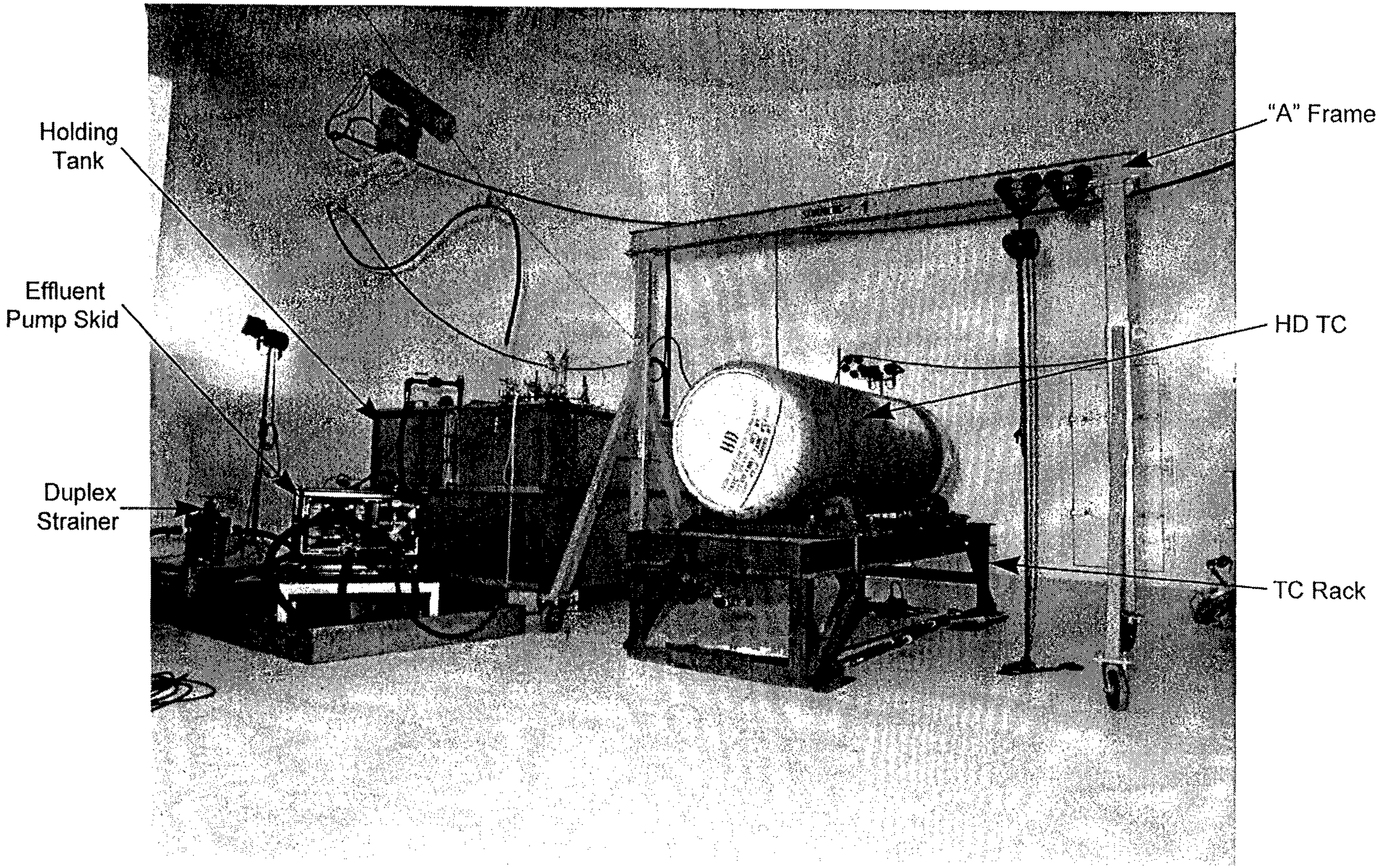


Figure 3-2. Demonstration Setup for the Second HD TC Cleanout Demonstration

The demonstration consisted of the steps listed below:

- Preparation of a drained HD TC
- Initial characterizations
- Steam flushing of the eductor tubes
- Pressurized hot water spray to remove heel
- Characterizations and vapor sample
- Pressurized hot water spray to achieve 3X
- Final characterizations and vapor sample

The HD TC was placed horizontally in a rack 3 feet off the chamber floor using an "A" frame and M1 Beam. The TC rack was tilted approximately 2 inches over its length to enhance draining. A ten inch hole was milled on the centerline of the TC in the end opposite the valves and eductor tubes using a modified milling machine that was mounted to the TC. The interface plate (Figure 3-3) was secured to the TC with four J-bolts and knobs. It sealed the opening to prevent any splashback and to hold the spray lance and drain line in place. In addition, the interface plate housed a pressure relief valve (set to relieve at 4 psig) and a vacuum breaker. The interface plate also allowed for (manual) linear movement of the spray lance along the axial length centerline of the TC.

The HD TC was cleaned with pressurized hot water supplied by a series of hot water generators placed outside the chamber. The Hydroblaster hot water generator was the main unit. A second hot water generator, either the Lanada unit or the M17 Decontaminating Apparatus, was used as a pre-heater. During hot water spraying operations, tap water from the chamber was supplied to the "pre-heater" hot water generator. The Lanada hot water generator (or the M17 Decontaminating Apparatus when the Lanada unit was not operating) was set at approximately 135°F. The effluent water from the pre-heater was directed to large tub or "pond". The temperature of the "pond" remained at approximately 125°F. The main hot water generator pumped the water from the pond and heated it to approximately 194°F. Prior to reaching the main hot water generator, the water passed through a 100 mesh filter which was attached to the hot water generator hose. For steaming operations, a second unit was not used. The necessary settings were adjusted on the Hydroblaster unit and steam was easily reached.

Block and check valves were installed in the hot water discharge line just inside the chamber wall to prevent flow from the chamber to the outside area. The hot water discharge line was connected to a spray lance and nozzle assembly.

The spray lance (wand) and nozzle assembly, as shown in Figure 3-3, included two rotating high pressure water jetting nozzles. The external end of the wand had an air motor drive, which allowed the nozzles to rotate, and oil mister. A movable support stand provided support for the overhung weight of the wand shaft and air motor

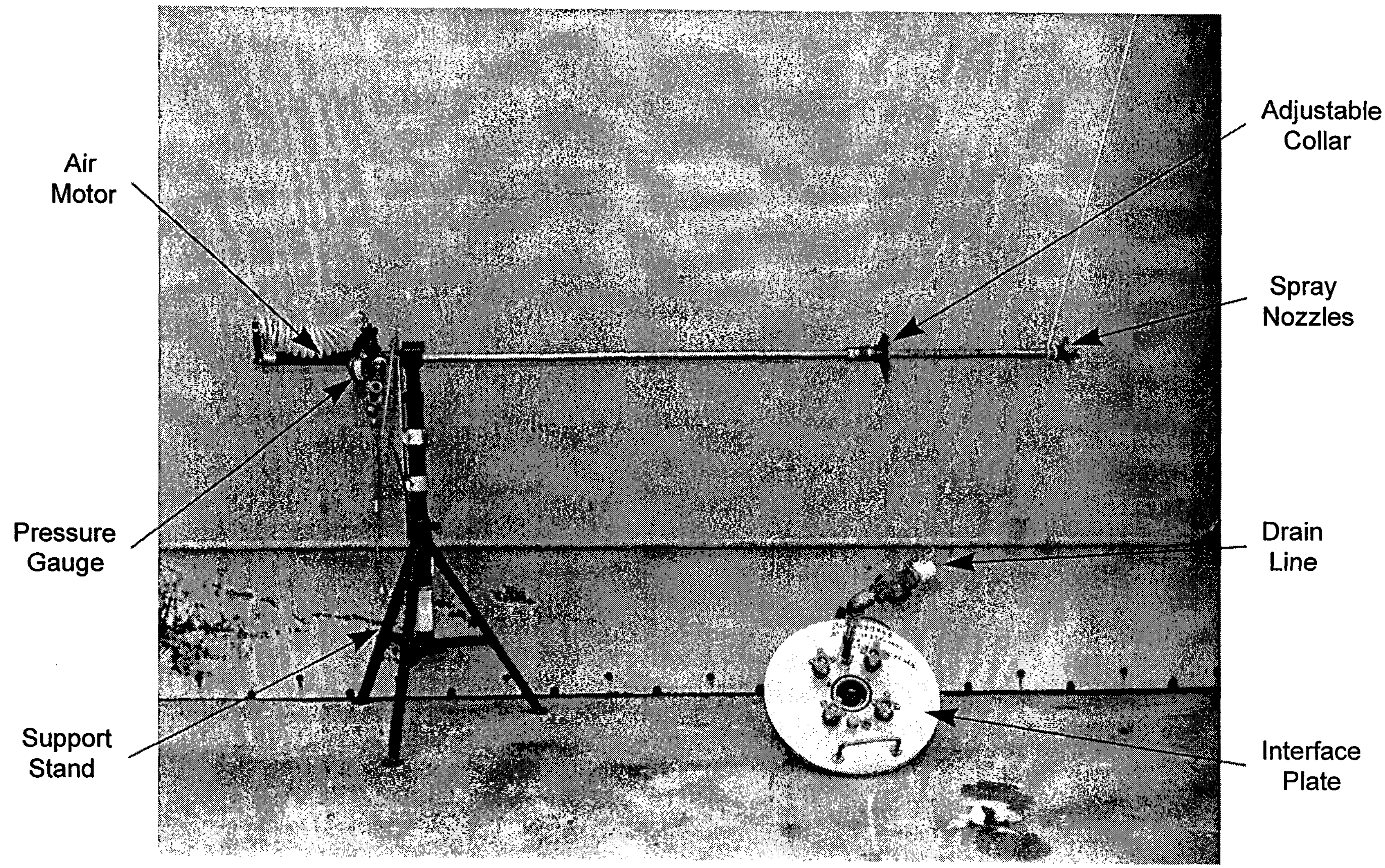


Figure 3-3. Lance and Nozzle Assembly & Interface Plate

assembly. The spray lance and nozzle assembly had provisions for adjusting the nozzle angular orientation. Angular position "0" was the initial position for the spray lance, which, when the spray nozzle assembly went through its rotation cycle, produced crosshatches approximately 1 inch apart. The spray patterns produced from one rotation are shown in Appendix D. Rotating the spray lance to angular positions 1, 2, and 3, provided angular increments on one-fourth the normal pitch, therefore, narrowing the crosshatches to approximately 1/4 inch apart. If necessary, the angular orientation of the lance was to be adjusted to ensure complete spray coverage of the interior surface. The crosshatches produced by the rotation cycles at angular positions 0 and 1 are also shown in Appendix D. For this demonstration, the angular position was kept at position "0".

The nozzles were also able to be located at different positions along the axial length centerline of the TC (the one-quarter, one-half, and three-quarters positions). For this demonstration, the one-quarter and the three-quarter positions were used since these positions corresponded to the approximate position where the nozzles would be located during the ton container cleanout process in the pilot plant design. The one-quarter and three-quarters positions are shown in Figure 3-4.

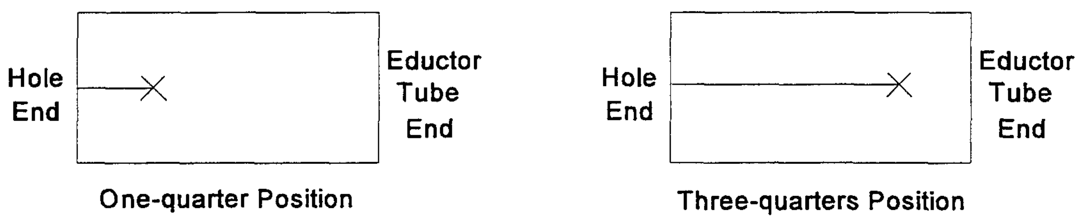


Figure 3-4. Nozzle Positions During Hot Water Spraying

Twelve skin thermocouples were mounted at designated paint-free locations on the exterior surface of the TC to monitor its skin temperature. A skid mounted suction pump system (Figure 3-5) contained the suction pump for draining the TC, hoses for plumbing the duplex strainer into the pump suction line, connections to permit back-flushing of the suction hose should it be necessary, and an air manifold. The drain line consisted of flexible ethylenepropylene dimonomer (EPDM) hoses between the TC and the pump and contained a thermocouple and a sight glass. A compressed air line (regulated to 125 psig) was connected to the drain line between the TC and the coarse strainer to backflush the line (back to the TC) if it became clogged.

The effluent pump was an ALL-FLO KN-10 air diaphragm pump constructed of Kynar with Teflon diaphragms that was capable of pumping water at a rate of up to 33 gpm using a maximum air pressure of 70 psi. During the TC cleanout, the effluent pump had failed. It was replaced by a Wilden M2 air diaphragm pump.

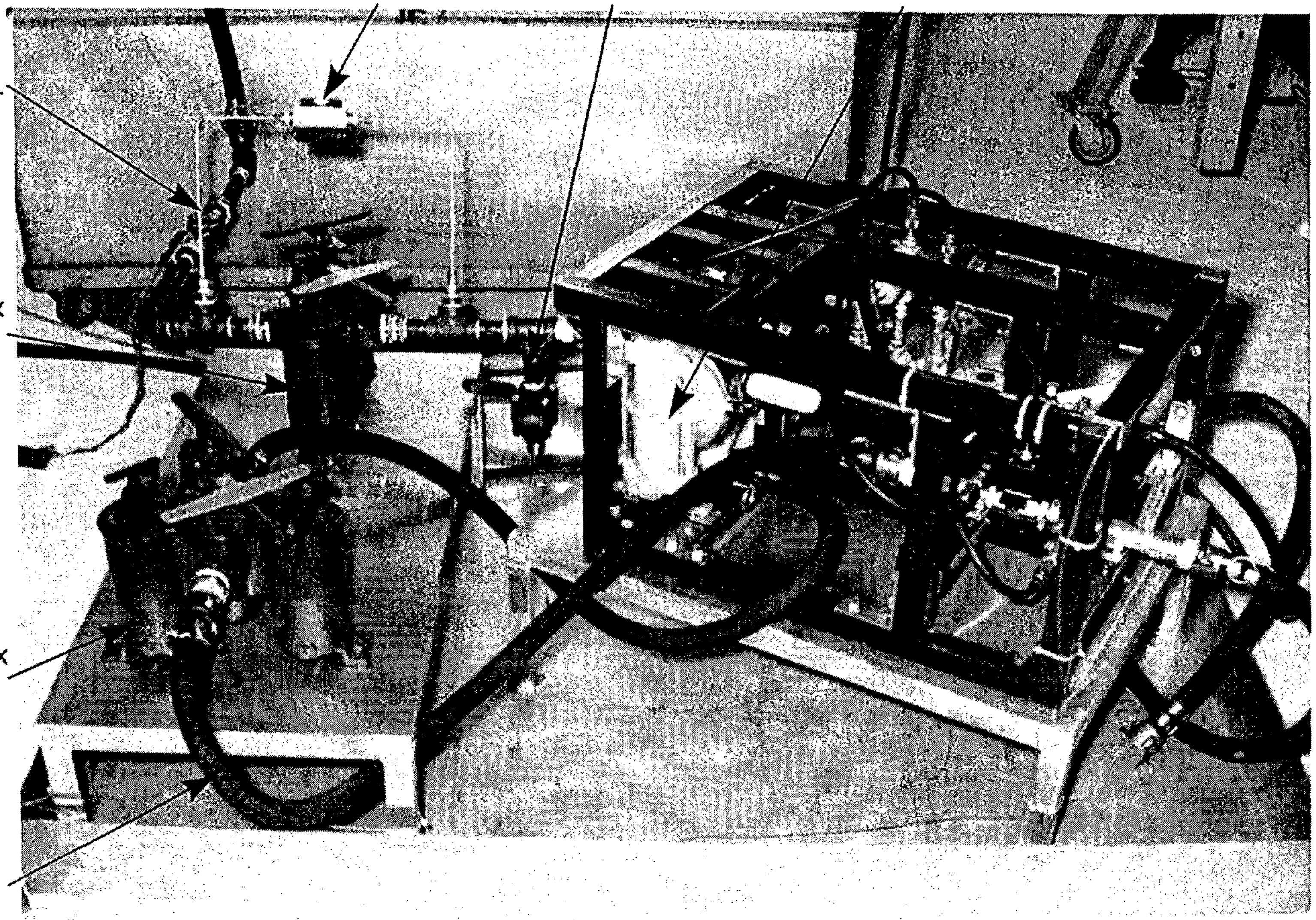


Figure 3-5. Pump Skid and Duplex Strainers

Cleanout effluents were strained with a coarse (1/4 inch perforation) duplex strainer ahead of the pump and a fine (80 mesh) duplex strainer after the pump (Figure 3-5). The strainers were Hayward Model 50 1-1/2 inch National Pipe Thread (NPT) duplex strainers constructed of cast iron with 316 stainless steel (SS) perforated or mesh baskets. The coarse strainer served to protect the air diaphragm pump, which could only handle solid particles less than 1/4 inch in diameter. The fine strainer served to keep smaller particles out of the holding tank and to assist in determining whether or not the effluent can be recycled.

After the first 30 minutes of hot water spraying, when the heel was removed, a settle tank and a Wilden M2 air diaphragm pump ("settle pump") were installed between the coarse strainer and the effluent pump. The stainless steel settle tank, as shown in Figure 3-6, was manufactured by the ERDEC machine shop. The settle tank contained a weir, which was located 1 foot from the inlet. In addition to inlet and outlet ports, a vent and a sample drain line were present.

The strained liquid effluent was pumped to a 1000 gallon, 316 stainless steel holding tank during the first 30 minutes of hot water spraying. The lines between the pump and the holding tank consisted of EPDM flexible hoses and carbon steel (American Society for Testing and Materials [ASTM] A53 black iron) pipe. During the second 30 minutes of hot water spraying, the effluent was pumped to the 600 gallon decon tank since the holding tank was leaking.

Vapor samples were taken with SUMMA® canisters from one of the TC plug holes (initial sample) or from the vent line off the hold tank (cleanout samples). The SUMMA® canisters are 6 liter evacuated spheres designed to collect a fixed volume of gases or vapors. For the holding tank samples, two gas sampling pumps, one set at 100 ml/min for the 1 hour sample and one set at 34 ml/min for the 3 hour sample, fed a constant flow rate of vapors to the SUMMA® canisters. A sampling pump was not used for the initial vapor sample from the TC; instead, the valve to the SUMMA® canister was opened and a sample was immediately collected. Methanol impingers were not used to collect a vapor sample during this demonstration since it was determined that the vapor sample collected in the SUMMA® canister would be sufficient.

For the eductor tube flushing, which was performed prior to the hot water spraying, the nozzle and wand assembly were installed to seal the ten inch hole and to prepare for the next stage of cleaning. A "Y wand" hookup (comprised of EPDM hoses, fittings, and two valves) were connected to the inlet of the eductor tubes. This equipment allowed the steam to pass through each eductor tube separately. The "Y wand" hookup is similar to the setup for the TC shown in Figure 3-7; however two valves were later installed to this apparatus to allow the flow of steam through each eductor tube separately.

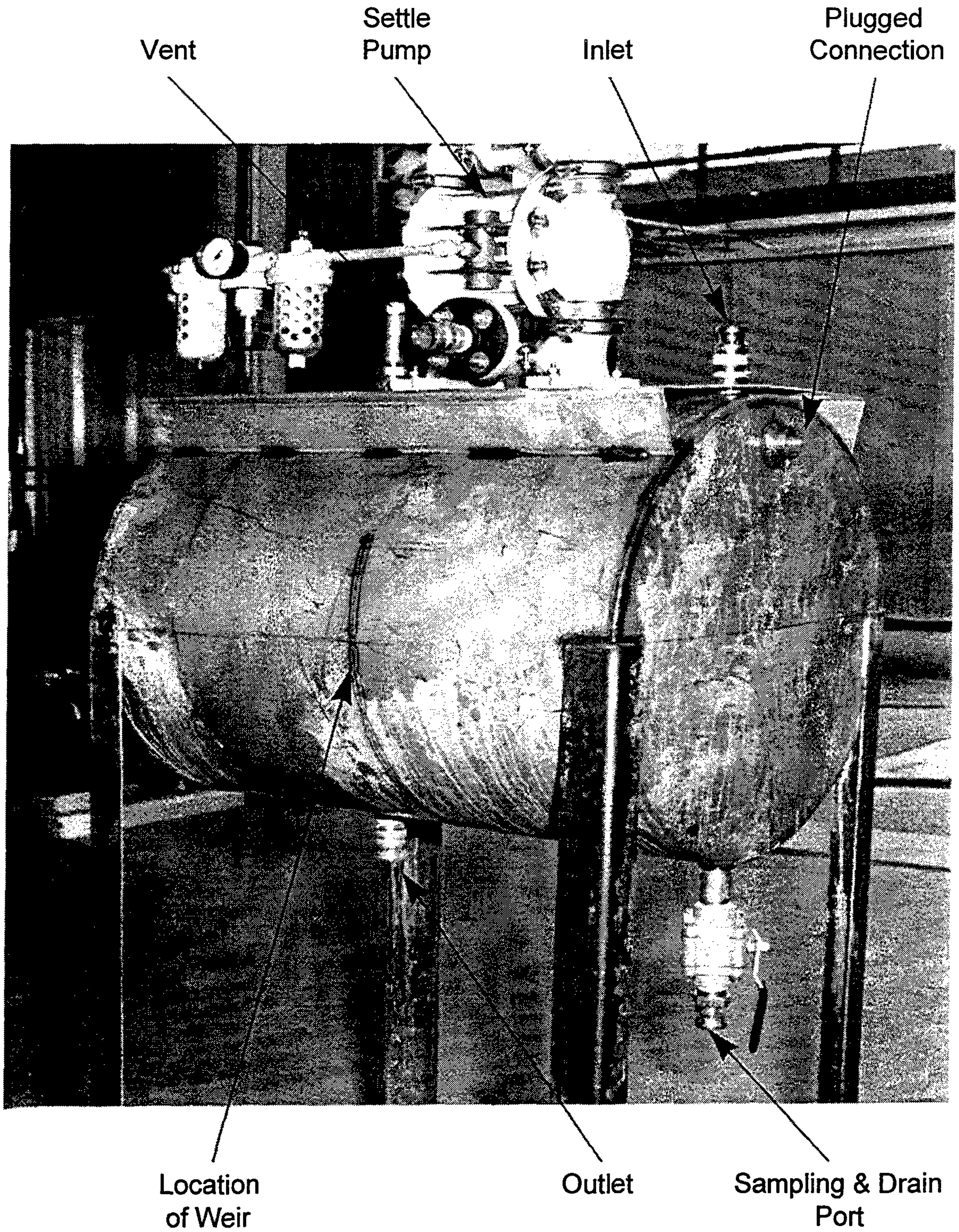


Figure 3-6. Settle Tank

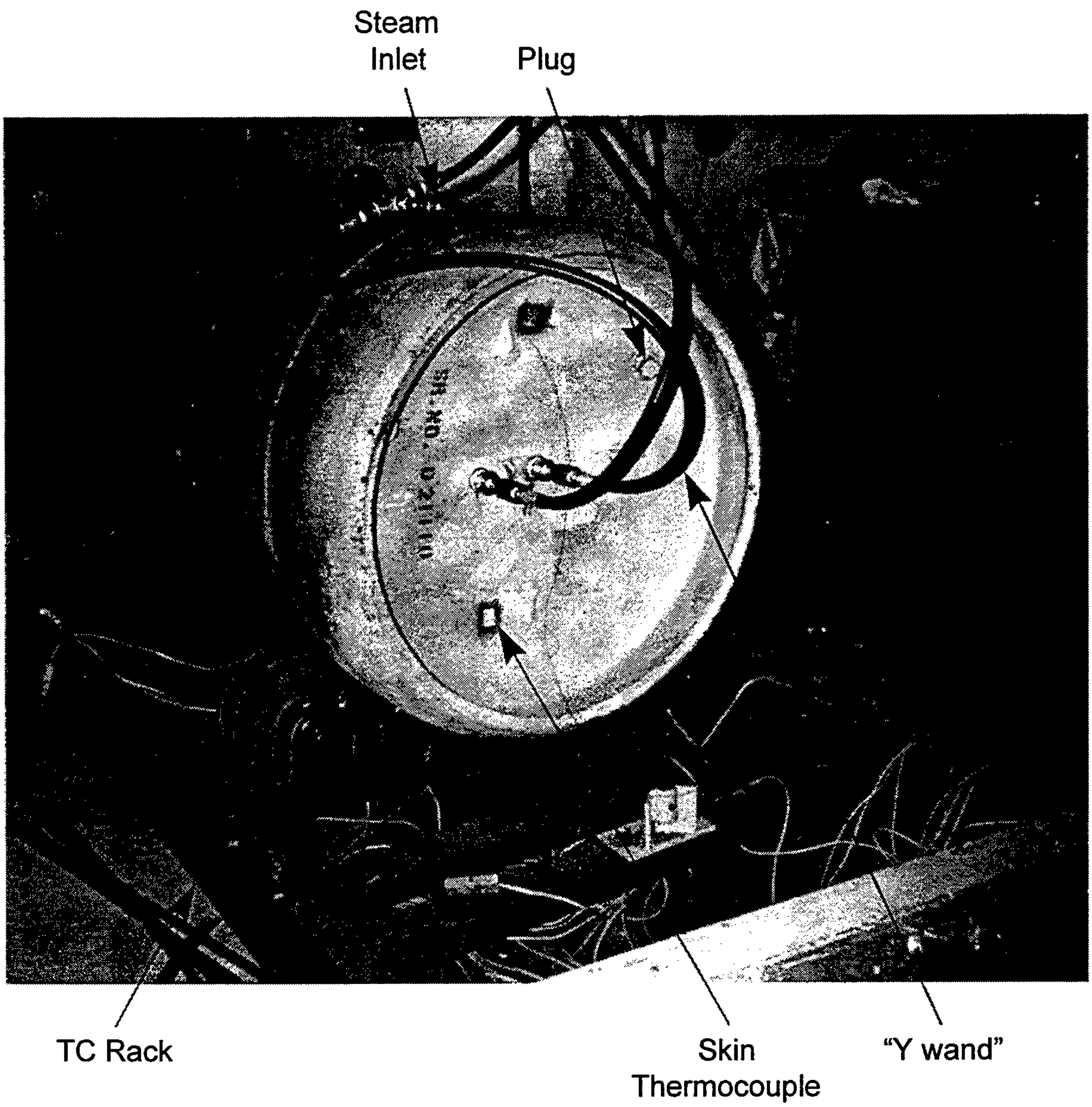


Figure 3-7. Setup for Steam Flushing of Eductor Tubes

The following data were collected during pressurized hot water spraying:

- Temperature, pressure, and flow rate of the hot water
- Surface temperature of the TC
- Temperature and flow rate of the effluent
- Duration of the pressurized hot water cleaning operation
- Amount of hot water used
- Amount of effluents generated

3.4 Demonstration Procedures

The second HD Ton Container Cleanout Demonstration was performed following the Standing Operating Procedure (SOP) entitled "Ton Container Cleanout" (Reference 8) and by following the Demonstration Change Form, dated 3 July 96, to the HD Ton Container Cleanout Demonstration Plan. The Demonstration Change Form can be found in Appendix C and includes the specific demonstration procedures. A summary of the demonstration procedures follows.

3.4.1 Preparation of a Drained HD TC

Record nameplate information. Determine weight of drained TC. Take intital vapor sample from TC. Cut ten inch hole in the center of the TC.

3.4.2 Removal of Solids

3.4.2.1 Initial Characterization. Inspect TC. Determine weight of TC. Obtain solid samples. Install interface plate and spray lance and nozzle assembly.

3.4.2.2 Eductor Tube Flush to Remove Heel in Tubes. Remove valves. Install steam flushing equipment. Flush each eductor tube with steam. Remove steam flushing equipment.

3.4.2.3 Hot Water Spray to Remove Heel (First 30 minutes). Spray interior of TC with pressurized hot water for 30 minutes. Allow TC to fill up for 10 minutes prior to starting effluent pump. Obtain vapor samples from holding tank. Collect liquid and solid effluent samples. Recirculate holding tank contents and obtain sample.

3.4.2.4 Characterization and DAAMS. Inspect interior of TC. Monitor the level of HD vapor contamination with DAAMS tubes if the interior of the TC is visibly clean.

3.4.3 Decontaminate TC TO 3X

3.4.3.1 Hot Water Spray to Achieve 3X (Second 30 minutes). Install settle tank and settle tank pump. Spray interior of TC with pressurized hot water for 30 minutes. Collect liquid and solid effluent samples.

3.4.3.2 Characterization and DAAMS. Inspect interior of TC. Monitor the level of HD vapor contamination with DAAMS tubes.

3.4.3.3 Hot Water Spray to Achieve 3X (Additional 30 minutes). Spray interior of TC with pressurized hot water in 30 minute intervals until interior of TC is determined to be in a 3X condition. When TC is in 3X condition, obtain settle tank sample. Recirculate the contents of the holding tank and obtain a sample. Inspect the interior of the TC. Weigh the TC.

3.5 Demonstration Results

3.5.1 Preparation of a Drained HD Ton Container

In July 1996, the TC (Serial #D94102) selected for the second HD TC Cleanout Demonstration was drained of mustard at the CTF. Based on a tare weight of 1600 pounds, it was estimated that there were approximately 1042 pounds of agent inside the TC. However, only 758 pounds were actually removed. This corresponds to 72 gallons of mustard. A sample (HD-S-6176-CTF-N) was collected for analysis.

The information on the ton container was:

Lot Ed 1881-1-K655
Tare Wt - 1600
Serial #D94102
1042.22 lbs

The information on the nameplate was also recorded and included:

Forge Welded Pressure Tank
American Welding Company
Carbondale, PA

The TC was then weighed with a Wagner Instruments load cell. The load cell was initially charged overnight to ensure an accurate measurement. First, the M1 Beam was weighed. A weight of 160 pounds compared closely with the M1 Beam weight determined in the previous demonstrations (158 lbs). The TC and the M1 Beam weighed 2114 pounds; therefore, the weight of the TC was 1954 lbs. The bonnet was then removed and weighed. The bonnet weighed 12.68 pounds. It was estimated that there were approximately 341 lbs of heel inside the TC based on a TC tare weight of 1600 pounds and the valve bonnet weight. The initial orientation of the TC on the rack was similar to how it was stored at the CTF. The TC was stored such that the valves and eductor tubes were in the vertical position, as shown in Figure 3-8.

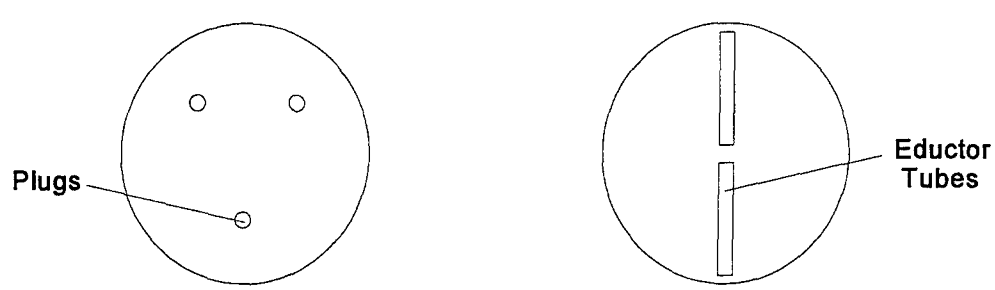
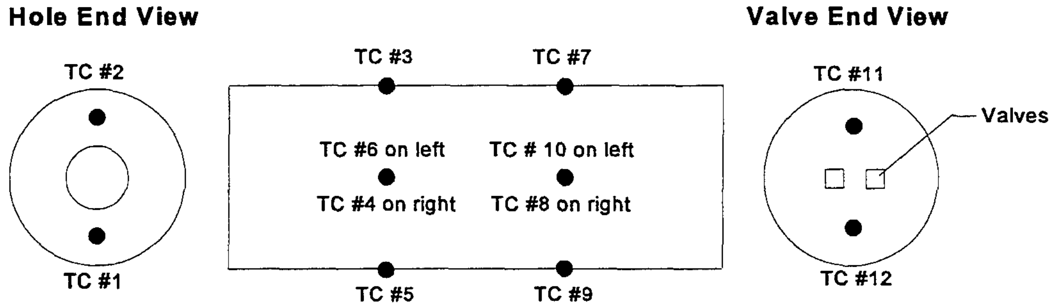


Figure 3-8. Orientation of Plugs and Eductor Tubes during Storage at CTF

On 10 July 1996, twelve skin thermocouples were installed on the TC. The twelve areas were first scraped to bare metal, the thermocouples were attached, and were functionally checked via the computer data acquisition system. The thermocouples were installed at locations as indicated in Figure 3-9. These locations represent the thermocouple locations after the TC was rotated 90 degrees to ensure that the heel was not at the bottom of the TC during the cleaning operations.



Note: Eductor Tubes were horizontal during TC cleaning.

Figure 3-9. Thermocouple Locations

Also on 10 July 1996, a benchmark vapor sample was taken from the hold tank using a 6 liter SUMMA® canister (Sample #C-03-02-HH-1926). A vapor sample (C-03-03-HH-1936) of the TC headspace was collected on 11 July 96 using another SUMMA® canister. A summary of the SUMMA® canister samples can be found in Table 3-1. After the vapor sample was taken, a flush mounted plug was installed. The remaining two plugs on the end of the TC to be cut were removed and flush mounted plugs were installed. The weight of the three plugs was 1.18 pounds.

The milling machine was mounted to the TC and a ten inch hole was milled on the centerline of the TC in the end opposite the valves and eductor tubes. It took approximately nine minutes to cut through the TC. The ten inch cutout did not fall into the TC, as with the first HD TC Cleanout Demonstration, but leaned inward on a slight angle. The milling machine was removed from the TC and the ten inch cutout was removed. The HD heel reached the bottom of the ten inch hole and prevented the cutout from falling into the TC. The removed cutout was weighed and set aside for examination and decontamination for disposal. The weights of the ten inch hole cutout and the metal filings were 17.56 and 1.82 pounds, respectively. After the cutout was removed from the TC, the MINICAMS®, which were being used to monitor the agent levels inside the Toxic Test Chamber, became saturated due to the extremely high levels of agent inside the chamber.

Table 3-1. SUMMA® Vapor Samples

Sample #	Sample Type	Time	Description
C-03-02-HH-1926	Benchmark SUMMA®	0	Taken off hold tank prior to adding cleaning TC & adding effluent to tank.
C-03-03-HH-1936	Initial SUMMA®	0	Taken through plug hole prior to cutting TC hole & cleaning TC.
C-03-13-HH-1986	60 min SUMMA®	60 (planned)	Lost (due to SUMMA® canister leak)
C-03-16-HH-1986	180 min SUMMA®	180 (planned)	Lost (due to SUMMA® canister leak)

3.5.2 Initial Characterizations

The TC interior was characterized using a camera and video recorder. Photographs were taken with a disposable camera and a hand-held video camera was used to inspect the interior of the TC through the cut hole. The TC was positioned such that the bottom of the TC as it was stored at the CTF, was at the 6:00 position.

The heel appeared similar to the heel from the first TC; however, there was much more heel in the current TC. The heel reached the bottom of the ten inch hole and covered almost three-quarters of the bottom eductor tube. The depth of the heel was estimated to be approximately 8 to 9-1/2 inches. The heel covered the TC from the 4:00 to 7:00 positions. The heel consisted of brown or black colored clumps or chunks similar to stalagmites. These clumps or chunks were extremely hard; in fact, it took significant effort to break off a piece while trying to obtain a sample. There was no observable liquid in the TC which was different than the first demonstration. It appeared that all of the liquid was successfully pumped out of the TC during the draining process. Air operated metering pumps were used to drain the TC. As with the first HD TC, a fill line was seen; however, for this TC, two fill lines were seen. One was approximately three-quarters up the TC and was more apparent. This dark brown to light brown line appears to be where the TC was originally filled. There was another fill line approximately halfway up the TC and was more of a rust-to-tar line. It appears that this line was the most recent agent level. The original estimate of HD in this TC, by the CTF, indicated that this TC was approximately half full. A photograph of the interior of the TC prior to cleaning can be found in Figure 3-10.

Figure 3-10. Interior of HD TC Prior to Cleaning

Six solid samples were removed from the TC using a scoop with a long handle and placed into 500 milliliter (ml) sample jars. Three were sent to the Analytical Chemistry Team (ACT) for analysis and three were sent to the CTF for storage until they were needed for further analysis. The characterization samples are summarized in Table 3-2.

Table 3-2. Characterization Samples

Sample #	Sample Type	Weight	Description
C-03-04-HH-1936	Scoop	0.27	Top of peak, 5:00 - 5:30 position
C-03-05-HH-1936	Scoop	0.48	Bottom of heel, close to wall, 5:00 position
C-03-06-HH-1936	Scoop	0.65	Peak pushed away, middle part sampled; 6:00 position
C-03-07-HH-1936	Scoop	0.64	Top of peak, 6:00 position
C-03-08-HH-1936	Scoop	0.78	Bottom of heel, close to wall, 7:00 - 7:30 position
C-03-09-HH-1936	Scoop	0.58	Peak pushed away, middle part sampled; 6:30 position

The scribing plate was placed over the ten inch hole and a circle was scribed on the TC end to ensure proper mounting of the interface plate. The interface plate was then secured to the TC.

In order to prepare for the steam flushing and hot water spraying operations, the TC was rotated 90 degrees such that the "CTF bottom" was located at the 3:00 position on 12 July 1996. This was done to ensure that the heel was on the side for easier cleaning and draining from the bottom. The three inch coverplate was installed to the interface plate to seal the TC.

On 15 July 1996, the agent concentration levels in the chamber had reduced to approximately 0.35 time-weighted-average (TWA). At 13:05 on 15 July 1996, the three inch coverplate for the interface plate was opened to determine if the heel had fallen to the bottom. The chamber technicians looked through the three inch hole with a flashlight, and observed that the majority of the heel was still at the 3:00 position. Only some of the loose chunks had fallen to the 6:00 position.

3.5.3 Steam Flushing of the Eductor Tubes

The lance and nozzle assembly was installed into the TC and the nozzles were located three-quarters into the TC (3/4 position, Figure 3-4). The wand was set to its baseline angular position, position "0". The three plugs from the eductor tube and valve end of the TC were removed and weighed a total of 1.22 pounds. One plug was steel, while the other two were brass. A vent line (EPDM hose) was connected from one of the threaded plug holes (on the valve end) to the holding tank. The hose was placed in the holding tank by inserting the hose under the lid. Temporary plugs were installed in the other two threaded holes. A benchmark sample of the tap water (C-03-01-HH-1986) from the "pond" of the hot water generators was taken for analysis.

The two valves were removed and the "Y wand" hookup for the steam was installed. On 16 July 1996, the eductor tube flushing was initiated. The temperature profile during the steam flushing of the eductor tubes can be found in Figure 3-11. On this plot, the first temperature spike was the pre-flushing steam verification check, and the second and third temperature spikes were the flushing of each eductor tube.

At 09:25, the steam generator was turned on, and when steam was obtained outside the chamber five minutes later, the steam was directed into the chamber. The chamber technicians allowed the steam to flow into a drum until steam, once again, was verified. Approximately 6 gallons of steam condensate was collected in the drum. The inlet temperature was 293°F. At 09:38, the steam line was hooked up to the "Y wand" hookup and the steaming of the right eductor tube commenced. The valve on the other part of the "Y" was closed so that the steam was only directed through one eductor tube at a time. The inlet temperature to the TC varied between approximately 175°F and 300°F. The steam pressure at the steam generator read 500 psi, while the steam pressure to the TC inlet was approximately 250 psi. Based on previous calculations, it was estimated that the flow of steam was approximately 1 gpm. The TC skin temperature immediately started to increase. The average skin temperature of the TC was 100°F. The inlet temperature increased until the eductor tube was cleared, and then the inlet temperature began to decrease.

At 09:43, the steam was switched to the left eductor tube for five minutes of steaming. Once again, the temperature increased (to approximately 375°F) until the blockage was cleared. The average skin temperature increased to approximately 115°F. A total of 10 gallons of condensate was added to the ton container during the flushing of the eductors. A summary of flushing the eductor tubes with steam can be found in Table 3-3.

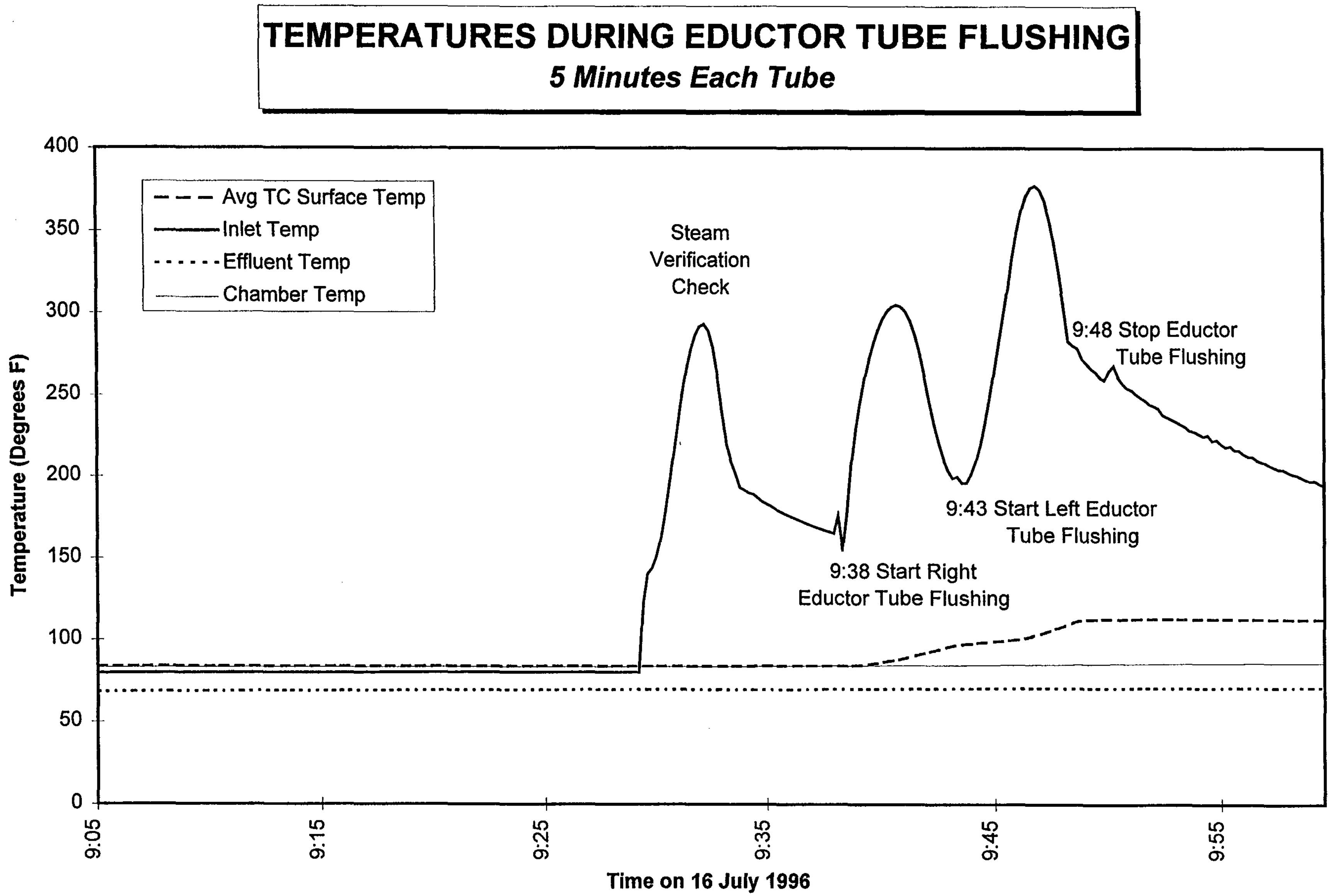


Figure 3-11. Temperatures during Steam Flushing of the Eductor Tubes

Table 3-3. Summary of Flushing Eductor Tubes with Steam

Parameter	Right Eductor Tube	Left Eductor Tube
Duration	5 min	5 min
Steam Flow Rate	1 gpm	1 gpm
Steam Pressure	250 psi	250 psi
Inlet Temp	175-300°F	200-375°F
Surface Temp	100°F	115°F
Total Amount of Condensate	5 gal	5 gal

3.5.4 Pressurized Hot Water Spraying To Remove Heel

3.5.4.1 First 30 Minutes of Pressurized Hot Water Spraying. The first 30 minutes of pressurized hot water spraying was initiated on 16 July 1996. The Hydroblaster and the Lanada hot water generators were operated in series. The Hydroblaster hot water generator, which was set to 240°F and 2550 psi, was used as the main unit and pumped water out of the "pond" supply. The Lanada hot water generator was used as a "pre-heater" and heated the water in the "pond" to 125°F. The pressure indicator at the wall (which had been determined to correlate closely with the pressure at the wand) was 2550 psi. Once the temperature reached the set temperature on the Hydroblaster unit (at 10:09), the pressurized hot water was supplied into the chamber, and thus, into the wand assembly. An inlet temperature of approximately 185°F and an inlet flow of 5 gpm were observed. Approximately 2.4 minutes into the spraying cycle (at 10:12), a connection to the hand-held wand, which was located outside the chamber failed and came apart from the hose. This failure had an immediate impact on the cleaning operation. The pressure at the wand decreased immediately to approximately 500 psi and the flow decreased immediately to 2.4 gpm. Also, the temperature rapidly decreased to 165°F. At the time of the failure, the skin temperature averaged approximately 125°F. A thermocouple located on the bottom of the TC reached 143°F. The low temperature, low pressure, and low flow water continued for another 3 minutes before the hot water generator was shut down (at 10:15). According to the inlet flow totalizer, approximately 17 gallons of water were introduced into the TC during these 5.4 minutes. The temperature and the flow rate profiles for the entire first 30 minutes of hot water spraying can be found in Figure 3-12 and Figure 3-13, respectively. Between the first and second parts of the first 30 minutes of hot water spraying, the computer data acquisition system was shut down, as indicated by the flat line on the plots. Also, a summary of the first part of the first 30 minutes can be found in Table 3-4.

Table 3-4. Summary of First Part of First 30 minutes of Hot Water Spraying

Duration	5.4 min
Pressure	1550 psi
Inlet Flow Rate	5 to 2.4 gpm
Inlet Temp	185 to 165°F
Surface Temp	130°F
Effluent Temp	N/A
Effluent Flow Rate	N/A
Total Amount of Water	17 gal

Since the plan called for a 10 minute soak time, no effluent was pumped out of the TC. Approximately 27 gallons of water (which included 10 gallons from the eductor tube flushing) remained in the TC. A small amount of liquid was seen trickling out of the bottom of the interface plate. The chamber technicians were forced to exit the chamber due to a combination of the extreme heat and length of time spent inside the chamber, and also due to equipment problems. The nature of these equipment problems are detailed in the following paragraph.

There were two hoses - one leading to the lance and nozzle assembly in the TC, (rated to 4500 psi) and a hose leading to a hand-held wand outside the chamber (rated to 3000 psi at 300°F). The hand-held wand was needed in parallel to the high pressure water line going into the chamber during the heat up and cool down of the hot water generators. During the subcontractor testing of the nozzle and wand assembly, it was determined that the optimum cleaning occurred in the range of 2400 to 2700 psi at the nozzle. There was a substantial pressure drop from the hot water generator to the nozzle, which measured, on the average, 700 to 800 psi; therefore, the hot water generator was set at approximately 3200 to 3500 psi to obtain the required nozzle pressure. The set pressure gauge on the hot water generator was used for information purposes only. In the past Ton Container Cleanout Demonstrations and during functional tests, no problems with the pressure were experienced. The failure did not seem to be associated with the pressure, but the wear and tear from the many hours of use that the hose had experienced. The hose to the hand-held wand was replaced with a spare hose, which was rated to 3000 psi at 300°F, since this was all that was available at the time.

3.5.4.2 Second Part of First 30 minutes of Hot Water Spraying. It was decided that the demonstration should be continued from where the hot water spraying stopped. It

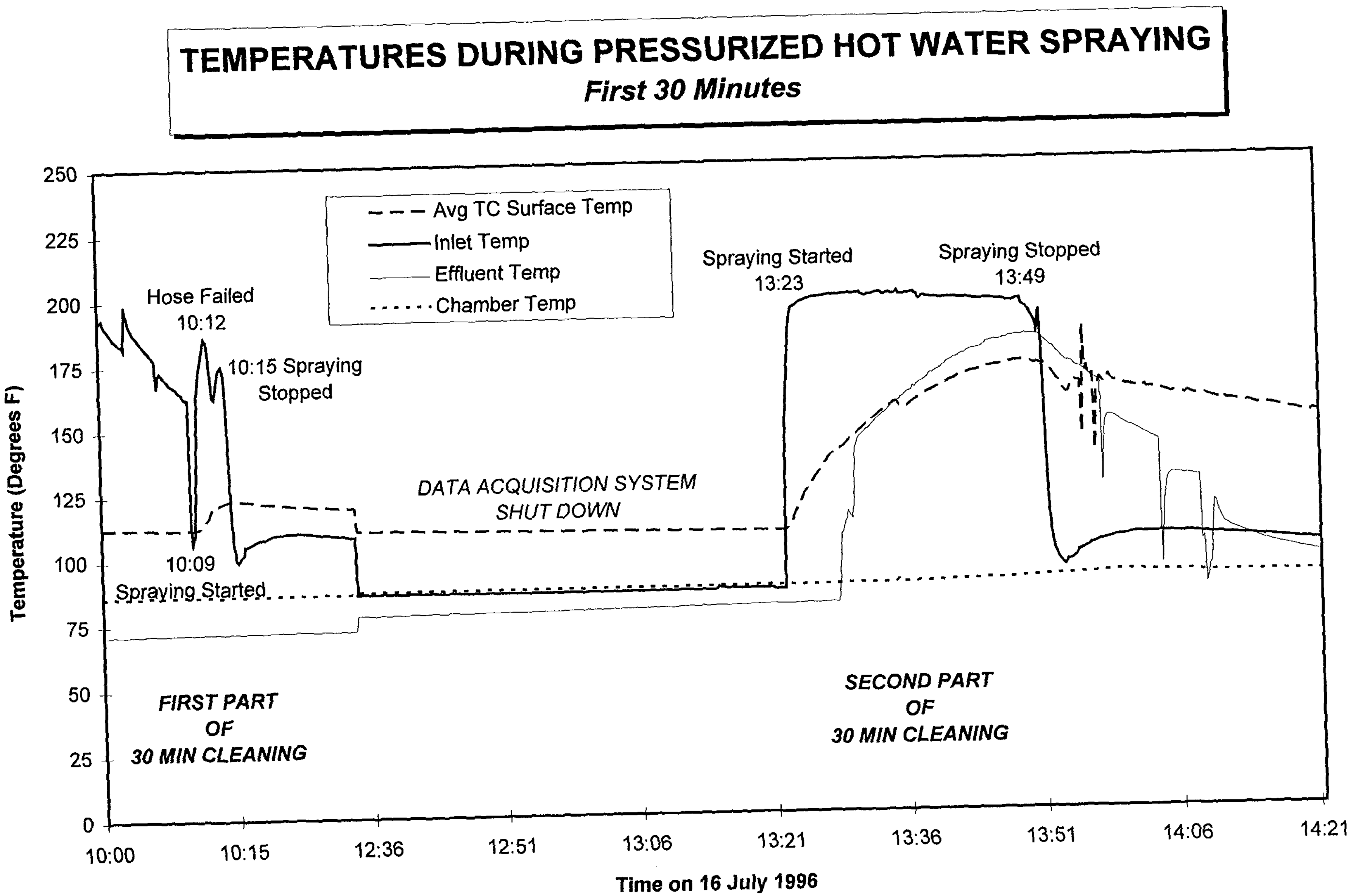


Figure 3-12. Temperatures during First 30 minutes of Hot Water Spraying

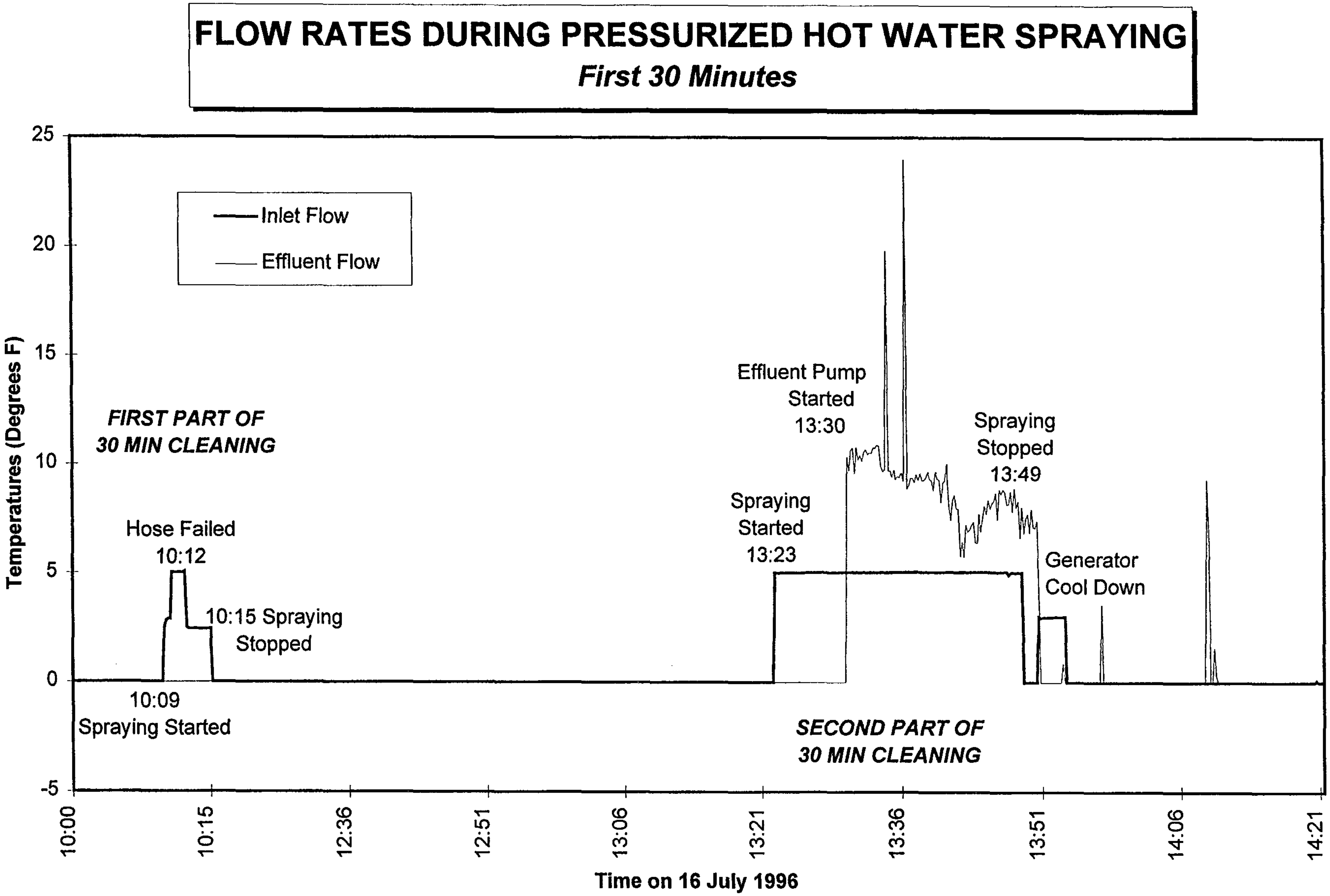


Figure 3-13. Flow rates during First 30 minutes of Hot Water Spraying

was desired to hot water spray the interior until 60 gallons of water had accumulated in the TC. At this time, 27 gallons of water was in the TC; therefore, 33 more gallons were needed to be sprayed before the effluent was to be pumped out. Based on a flow rate of 5 gpm, approximately 6.5 minutes were required to reach the 60 gallon total, which was an equivalent of 10 minutes of hot water spraying at 5 gpm.

The 27 gallons of water remained in the TC for 3 hours and 18 minutes. During this time, the average TC skin temperatures decreased from 130°F to 105°F. Also, during most of the down time (from approximately 12:30 to 13:20), the computer data acquisition program was shut down, as indicated by the flat lines in the temperature profiles in Figure 3-12. The chamber technicians entered the Toxic Test Chamber at 13:19 to continue with the first 30 minutes of hot water spraying. The hot water generators were started and at 13:23, the pressurized hot water was directed into the TC. The wand pressure was 2700 psi. After operating for 6.5 minutes, it was estimated the remaining 33 gallons of water had accumulated in the TC and the effluent pump was then started. This total amount of water was equivalent to 10 minutes of hot water spraying at 5 gpm (in addition to the 10 gallons from the eductor tube steam flushing). A brownish liquid sample (C-03-10-HH-1986) was collected at this time. The pH of this sample was between 0 and 1. No solids were found in the 80 mesh fine strainer; however, approximately 20 grams were collected from the 1/4 inch coarse strainer (Sample #C-03-11-HH-1986). A summary of these samples can be found in Table 3-5.

Table 3-5. Effluent Samples During First 30 minutes of Hot Water Spraying

Sample #	Sample Type	Time	Volume	Weight	Description
C-03-10-HH-1986	Liquid	10	400 ml	380 g	brownish liquid pH: 0-1
C-03-11-HH-1986	Coarse Strainer	10	...	20 g	solid
C-03-13-HH-1986	Liquid	20	400 ml	390 g	brownish liquid pH: 0.5
C-03-16-HH-1986	Liquid	30	350 ml	360 g	brownish liquid pH: 0-1

It was planned that the 60 minute and 180 minute SUMMA® canisters were to be opened as soon as the holding tank began collecting effluent. Valid samples could not be collected at this point; however, the SUMMA® canister valves were opened at the beginning of the next chamber entry (17:00). A vacuum was not observed and a

sample at this time was not collected in the SUMMA® since they were already filled. It appeared that a small leak had developed and the canisters lost their vacuum. The SUMMA® canisters had been evacuated to 25 inches prior to the test on 10 July 1996. A summary of the SUMMA® canister samples can be found in Table 3-1.

After 15 minutes (13:38) of cleaning, the chamber operators attempted to move the lance and nozzle assembly from the 3/4 position to the 1/4 position; however, the wand would not budge. There were areas of rust over many parts of the wand and it was difficult to loosen. It was decided to continue and keep the nozzle at the 3/4 position for the entire first 30 minutes and to move it to the 1/4 position at the end of this stage of hot water spraying.

At approximately 20 minutes (13:41), data were recorded and another brownish liquid sample (C-03-13-HH-1986) was taken. The pH of this sample was approximately 0.5. The strainers were not checked at this time.

At 30 minutes (13:49), data were recorded and another brownish liquid sample (C-03-16-HH-1986) was taken. As with the past two samples, the pH was between 0 and 1. The hot water generator was shut down at the end of 30 minutes of hot water spraying. No solids were found in either strainer but a greyish film was present on the screen. The effluent samples taken during the first 30 minutes of hot water spraying are summarized in Table 3-5.

According to the inlet flow totalizer, the total quantity of water introduced into the TC during this part of the first 30 minutes of hot water spraying was 135 gallons. The total amount of water used during the first 30 minutes of hot water spraying was 152 gallons (17 gallons from the first attempt and 135 gallons from the second attempt).

The temperature and flow rate profiles during the second part of the first 30 minutes of hot water spraying can be found in Figures 3-12 and 3-13. As previously stated, the computer data acquisition program was shut down from approximately 12:30 to 13:20, as indicated by the flat lines in the temperature profiles. The inlet temperatures remained relatively steady and ranged from 195 to 200°F. The inlet flow was constant at 5 gpm and the pressure at the wand was approximately 2700 psi. All the skin thermocouples reached approximately 185 to 190°F with the exception of two thermocouples (Thermocouple #1 and Thermocouple #11, see Figure 3-9). The thermocouple below the ten inch hole (Thermocouple #1) came loose when liquid began to leak out of the TC. The temperature of the thermocouple above the valves (Thermocouple #11) also did not increase significantly. The effluent temperature was 185°F toward the end of the cleaning cycle and the effluent flow ranged from 6 to 11 gpm. After the 30 minutes of cleaning, the thermocouple on the bottom of the TC closer to the hole end (Thermocouple #5) had increased to over 205°F. A summary of the second part of the first 30 minutes of hot water spraying can be found in Table 3-6.

Table 3-6. Summary of Second Part of First 30 minutes of Hot Water Spraying

Duration	26.5 min
Pressure	2700 psi
Inlet Flow Rate	5 gpm
Inlet Temp	195 to 200°F
Surface Temp	185 to 190°F (except 2 thermocouples)
Effluent Temp	185°F
Effluent Flow Rate	6 to 11 gpm
Total Amount of Water	135 gal

After the hot water generator was stopped after 30 minutes of hot water spraying, low flow, low pressure, low temperature water was unintentionally introduced into the TC during the cool down of the hot water generator. It was estimated that approximately 9 gallons of water at 3 gpm was introduced. This water accumulated in addition to the liquid that was in the TC that could not be completely pumped out. Over this 3 minute period, the inlet temperature decreased from 180 to 95°F. The pressure at the wand was close to 0 psi. At this pressure and flow, there was not a spraying effect and the water trickled out of the nozzle.

3.5.4.3 Recirculation of Holding Tank. After the first 30 minutes of hot water spraying, the demonstration plan called for the contents of the holding tank to be recirculated for 30 minutes and the appropriate samples be taken. Problems with the pump prevented the contents of the holding tank from being properly recirculated. As a result, samples were obtained from the hoses between the pump and the holding tank. The samples are listed in Table 3-7.

The 1000 gallon holding tank, which was made from stainless steel since it was originally planned to be used for a "one-time demonstration", developed leaks. This holding tank had been used for the first HD TC and the VX TC Cleanout Demonstrations. A large puddle was observed underneath the tank. On the morning of 17 July 1996, it was noticed that the contents of the 1000 gallon holding tank had emptied on the chamber floor due to the numerous leaks in the tank. The liquid was yellowish in color with a pH of 1. The chamber floor was then decontaminated and the TC rack, which held liquid that had leaked out of the TC, was drained and rinsed with water. The MINICAMS®, at this time, were saturated; and therefore, could not indicate the level of agent concentration in the chamber.

Table 3-7. Holding Tank Samples

Sample #	Sample Type	Volume	Weight	Description
C-03-19A-HH-1986	Holding Tank	250 ml	260 g	brownish liquid
C-03-19B-HH-1986	Holding Tank	300 ml	300 g	brownish liquid
C-03-19C-HH-1986	Holding Tank	400 ml	420 g	brownish liquid
C-03-19D-HH-1986	Holding Tank	2 gal	...	brownish liquid

3.5.5 Characterizations after 30 minutes of Hot Water Spraying

The lance and nozzle assembly and interface plate were removed so that the interior of the TC could be observed. Photographs were taken with a disposable camera and a video was made using a hand-held video camera. As expected, approximately 10 to 15 gallons of liquid remained in the TC. This liquid was clear and the chamber technicians could see the bottom of the TC through the liquid. No solids remained in the TC. The eductor tube wall was visibly cleaner. There was rust that looked like it had bubbled on the top part of the TC, mainly closer to the hole end. Areas of rust were also observed on the eductor tube wall and on the ends of the eductor tubes. A photograph of the interior of the TC after 30 minutes of hot water spraying can be found in Figure 3-14.

A liquid sample (C-03-F1-HH-1996) was removed from the TC to determine the pH and the HD concentration of the liquid that remained in the TC. The liquid was clear with a pH of between 2 and 3. A liquid sample (C-03-F2-HH-1996) was also taken from the a strainer using a syringe. The liquid in the strainer resulted from the partial recirculation of the holding tank. The pH of the strainer liquid was between 0 and 1. The external lip of the TC below the ten inch hole contained liquid and it was tested with M8 paper. Results indicated that no agent was present. These additional samples are summarized in Table 3-8.

Table 3-8. Additional Samples After 30 minutes of Hot Water Spraying

Sample #	Sample Type	Volume	Weight	pH
C-03-F1-HH-1996	TC Liquid	400 ml	420 g	2-3
C-03-F2-HH-1996	Strainer Liquid	300 ml	300 g	0-1

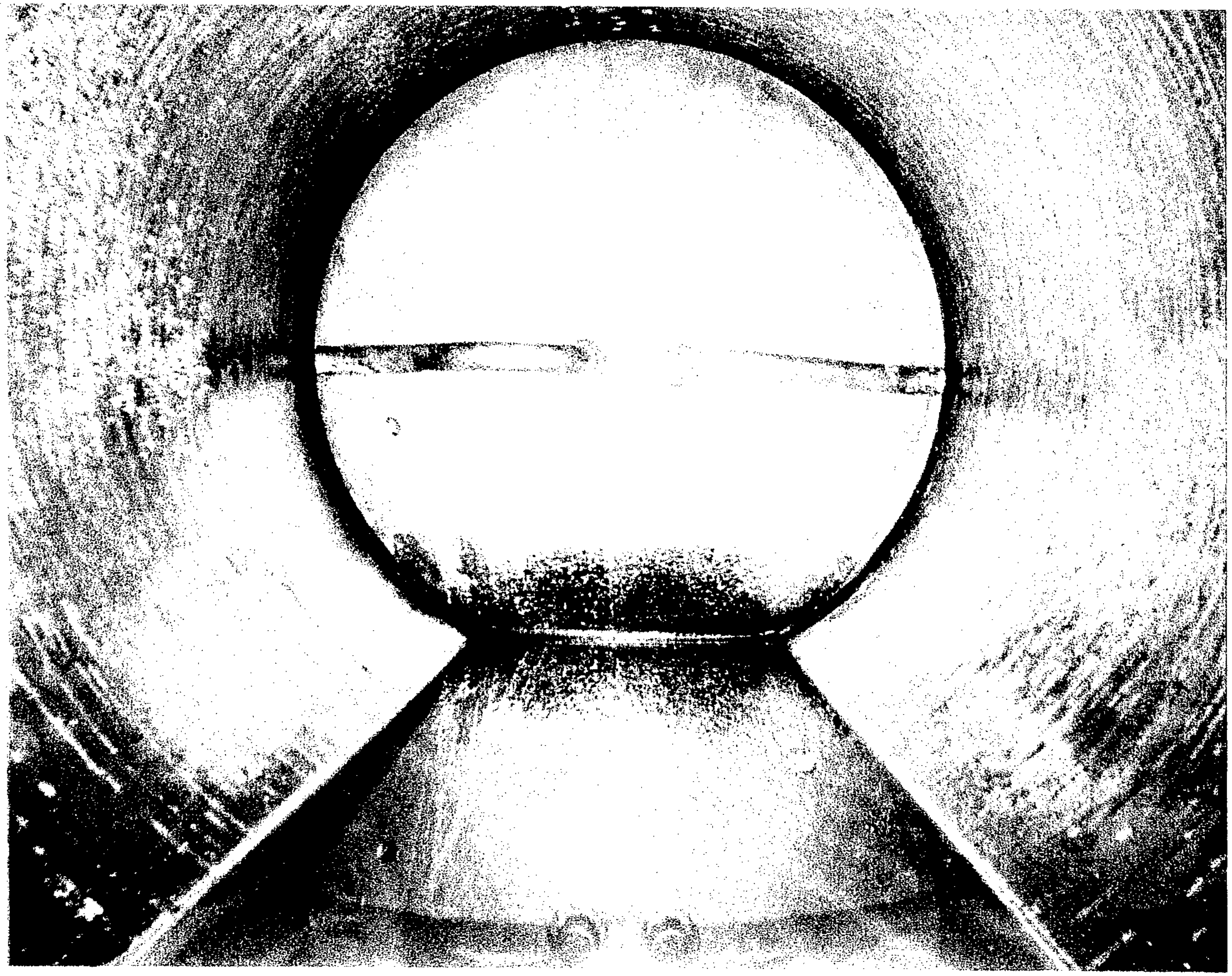


Figure 3-14. Interior of HD TC After 30 minutes of Hot Water Spraying

3.5.6 Second 30 minutes of Pressurized Hot Water Spraying to Achieve 3X

3.5.6.1 Preparation for Second 30 minutes of Hot Water Spraying. Due to equipment failures previously discussed, the second 30 minutes of hot water spraying were not able to be completed on the same day as the first 30 minutes of hot water spraying. Preparations for the second 30 minutes of hot water spraying began with the installation of a Wilden M2 air diaphragm pump to replace the effluent pump that had failed. The new "effluent pump" was functionally checked with a bucket of water and was used to pump the remaining liquid out of the TC.

The hot water generators were once again functionally checked. A hose rated to 300 bar at 175°C (which is equivalent to 4410 psi at 347°F) was installed and replaced the hose to the hand-held wand. Due to problems with the Lanada hot water generator, which was needed to preheat the water, an M17 Decontaminating Apparatus was obtained as a backup preheater.

The "Y wand" hookup for the steam was to be removed and two plugs were to be installed; however, the two plugs were not available. It was decided to leave the "Y wand" installed and keep the two valves on the "Y wand" closed.

The settle tank was installed and the necessary hoses were connected. As a result of leaks, the 1000 gallon holding tank was removed from service and replaced with the 600 gallon decontamination tank.

Prior to starting the cleaning operation, the 3 inch mounting plate was attached to the interface plate to seal the TC to determine if the agent concentration levels inside the test chamber had decreased. The MINICAMS® indicated that the chamber was still saturated with HD, and therefore accurate agent levels could not be obtained.

The 1000 gallon holding tank was rinsed with water and a high-test hypochlorite (HTH) bleach solution. This tank was also cleaned with pressurized hot water using the hand-held wand from the hot water generator. After cleaning the tank, the chamber was still saturated with HD; however, it was questionable as to whether the agent concentration was really high or the thiodiglycol was possibly interfering with the MINICAMS®. A DAAMS vapor sample of the chamber was then requested to determine if thiodiglycol was interfering with the MINICAMS®, or that the chamber was indeed saturated with mustard. Results from Sci Tech Laboratory indicated that "gross amounts" of mustard were present in the chamber. These results were confirmed by the mass spectrometer.

The TC was purged with "clean", dry air from the chamber's compressed air line. The interface plate was secured to the TC and the plate for the DAAMS tubes was installed in the 3 inch hole. One tygon tube was used for air inlet and one for the outlet. The TC purge was initiated at 20:11 on 23 July 1996 and stopped exactly 5 minutes later.

When the purge was started, approximately one cup of liquid came out of the outlet tube, possibly due to surface condensation. The TC was closed up and allowed to "off-gas" overnight. The temperatures varied from 73°F to 80°F, which met the minimum requirement of 70°F.

A DAAMS vapor sample (C-03-F3-HH-2066) was initiated at 08:45 on 24 July 1996 and the vacuum pump pulled a sample for the required 2 hours. Two DAAMS tubes were used: SPA003 and SPA005. The DAAMS tubes were analyzed by the EAI Sample Processing Area at E3726 and results indicated that the HD agent concentration was 434.8 micrograms/milliliter (μg/ml), which was equivalent to approximately 4.8 TWA based on the pump flow rate setting of 250 milliliters/minute (ml/min). The second tube was not analyzed since the first indicated that agent was present.

As stated earlier, there was a rusty film and corrosion over many areas of the wand which had prevented the operators from moving it during the cleaning process. The wand could not be moved while it was attached to the interface plate. The lance and nozzle assembly was removed from the interface plate and loosened using wrenches. The nozzle was moved to the one-quarter position (Figure 3-4).

Prior to initiating the second 30 minutes of hot water spraying, the entire system was, once again, functionally tested with water to ensure all equipment was working properly. All equipment functioned, except the Lanada hot water generator, which was then replaced with the M17 Decontaminating Apparatus.

3.5.6.2 Second 30 minutes of Hot Water Spraying. The hot water generators were started at approximately 15:00 on 24 July 96 and were allowed to reach the desired temperature. The set point on the Hydroblaster was adjusted from 240 to 280°F so the burner would not shut off when a temperature of 240°F was reached, which in previous tests, had caused the inlet temperature to drop to approximately 160°F. Since there was no set point on the M17 Decontaminating Apparatus, the fuel control was adjusted so that the water in the "pond" remained at approximately 125°F. The second 30 minutes of hot water spraying the interior of the TC (with the nozzle in the one-quarter position, see Figure 3-9) was initiated at approximately 15:14. The Hydroblaster unit pressure was 3550 psi and the pressure at the indicator on the wall outside the chamber, which has been determined to correspond closely with the wand pressure, was 2750 psi. Approximately two minutes later, the pressure on the wand was 2600 psi and the air pressure to the motor was 35 psi (33 to 37 psi was desired). Liquid was observed leaking out of the interface plate.

At 15:29, fifteen minutes of hot water spraying were completed. The necessary data were recorded and the samples were collected. The hot water generator pressure was set at 3600 psi, while the pressure at the wall was 2800 psi. The inlet temperature averaged 219°F at 5 gpm. The average TC skin temperature was 209°F. The effluent

flow was approximately 7 gpm and the effluent temperature was 220°F. No solids were observed in either strainer. A liquid sample (C-03-20-HH-2066) was taken. The pH was measured using pH paper to be between 4 and 5.

At 15:30, it appeared that the o-ring seal on the interface plate failed since a steady stream of steam was observed escaping from the TC (on the left side of the interface plate) for a few minutes. The water that had leaked out of the TC (through the bottom of the interface plate) dripped into the TC rack, and was tested with M8 paper. Results indicated that no agent was present.

At 15:44, thirty minutes of hot water spraying were completed. The inlet temperature was approximately 212°F and the inlet flow was approximately 5 gpm. The average TC skin temperature was 200°F. The effluent temperature was 196°F, while the effluent flow was approximately 4.5 gpm. No solids in the strainers were observed. The hot water generators were shut down and the water supply line to the wand was disconnected. The effluent and settle tank pump were operated until no flow was observed on the effluent. It took approximately two minutes to remove all liquid from the TC.

The temperature and flow rate profiles during the second 30 minutes of hot water spraying can be found in Figure 3-15 and 3-16, respectively. A summary of the second 30 minutes of hot water spraying can be found in Table 3-9. During this stage of cleaning, the inlet flow rate remained at approximately 5 gpm and the pressure of the wand was approximately 2600 psi. The inlet temperature cycled between 199 and 235°F. These temperatures, although slightly exceeding the desired range, were considered acceptable.

The average skin thermocouples, with the exception of the four thermocouples on the TC ends, reached approximately 212°F. The thermocouples on the TC ends (Thermocouples 1, 2, 11, and 12; see Figure 3-9), increased, at the most 10°F, to 105°F.

The effluent temperatures had reached 240°F during this stage of hot water spraying. At approximately 10 minutes into the cleaning cycle, the effluent temperatures exceeded the inlet temperature. This occurred for approximately 15 minutes before the inlet temperature once again exceeded the effluent temperature. The effluent flow rate ranged from 4.5 to 8 gpm. Also, the ambient temperature had increased from 80 to 87°F during this 30 minute stage of cleaning.

The lance and nozzle assembly, and the interface plate were removed from the TC. Approximately one cup of effluent was still left in the TC that was not removed. This liquid that remained in the TC was soaked up with a clean sponge. There was also liquid on the external lip of the TC (beneath the ten inch hole) that was soaked up as well.

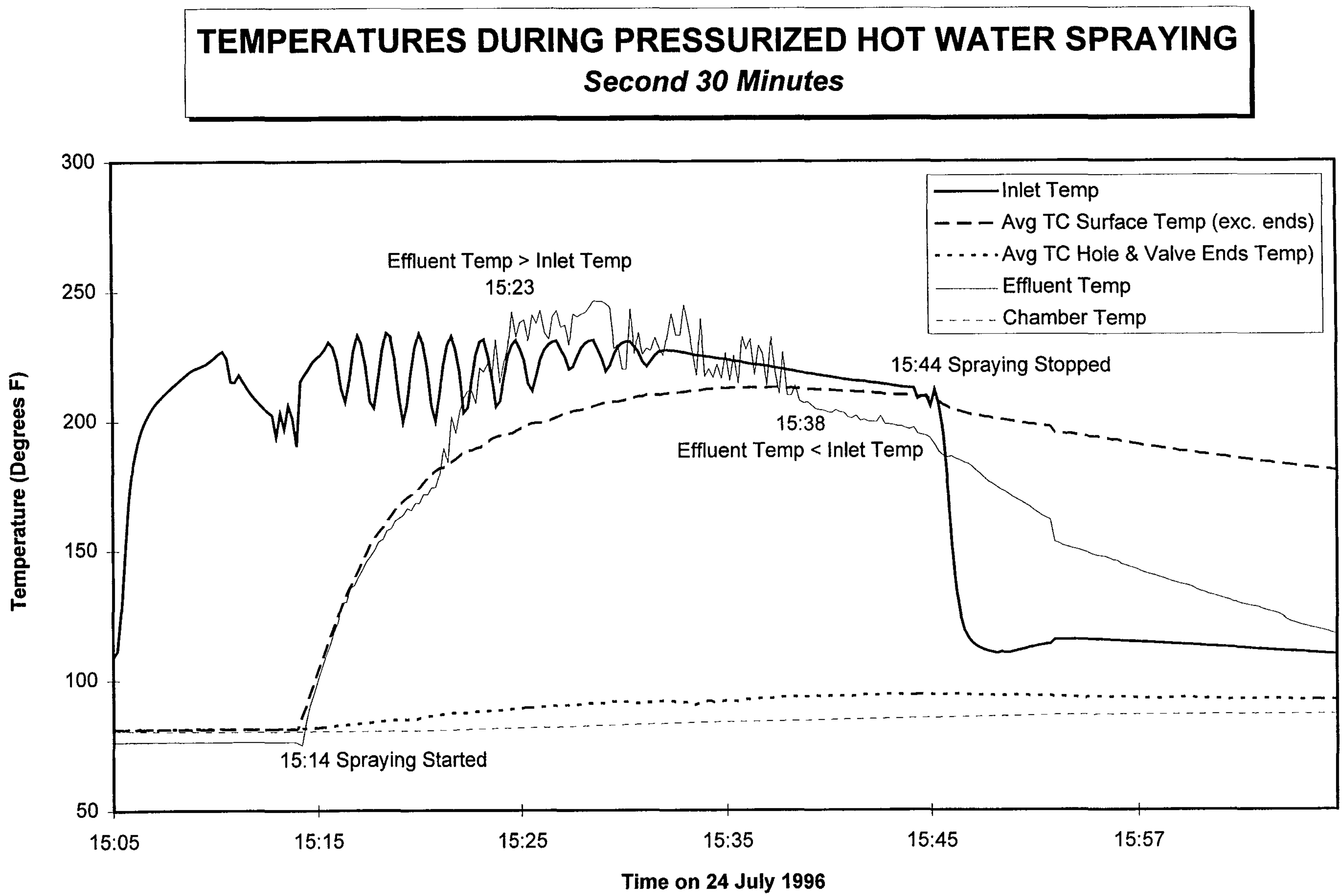


Figure 3-15. Temperatures during Second 30 minutes of Hot Water Spraying

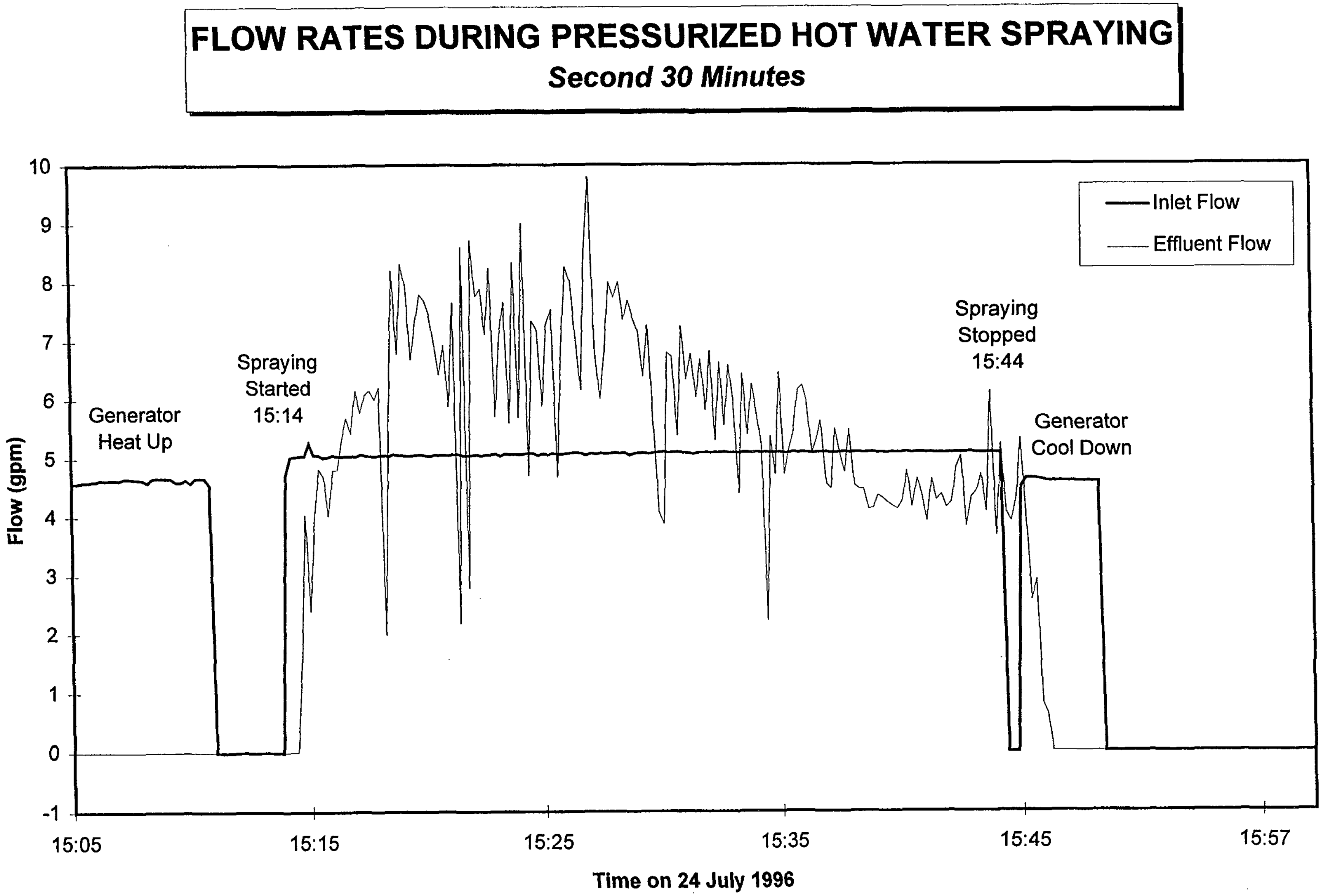


Figure 3-16. Inlet and Effluent Flows during Second 30 minutes of Hot Water Spraying

Table 3-9. Summary of the Second 30 minutes of Hot Water Spraying

Duration	30 min
Pressure	2600 psi
Inlet Flow Rate	5 gpm
Inlet Temp	199 to 235°F
Surface Temp	212°F (except TC ends)
Effluent Temp	240°F
Effluent Flow Rate	4.5 to 8 gpm
Total Amount of Water	153 gal

After the second 30 minutes of hot water spraying, the settle tank was full. A 350 ml sample (C-03-23-HH-2066) and a four gallon sample (C-03-F4-HH-2066) were taken for analysis. A 250 ml sample (C-03-F5-HH-2066) was taken from the four gallon sample for additional analysis. An additional sample (C-03-F6-HH-2066) was also taken in case it was needed for additional particle size distribution. A summary of these samples can be found in Table 3-10.

Table 3-10. Effluent Samples During & After Second 30 minutes of Hot Water Spraying

Sample #	Sample Type	Time	Volume	Weight	Description
C-03-20-HH-2066	Liquid	45	350 ml	370 g	clear liquid, black precipitate: pH: 4-5
C-03-23-HH-2066	Settle Tank	60	350 ml	380 g	dark grey/black liquid pH: 5-6
C-03-F4-HH-2066	Settle Tank	60	4 gal	...	additional sample
C-03-F5-HH-1986	Settle Tank	60	250 ml	250 g	250 ml sample from 4 gallon sample
C-03-F6-HH-1986	Settle Tank	60	350 ml	350 g	additional sample

3.5.7 Characterizations and Vapor Sample after 60 minutes of Hot Water Spraying

3.5.7.1 Characterizations. After 60 minutes of hot water spraying, a rusty film was observed over the entire cylindrical interior surface of the TC. The eductor tube wall was clean and appeared greyish. The "bubbled" rust was no longer present. Some black spots and score marks from the water pressure from the nozzle were able to be seen. Overall, the TC was much cleaner than after 30 minutes of hot water spraying. A photograph of the interior of the TC after 60 minutes of hot water spraying can be found in Figure 3-17.

3.5.7.2 Vapor Level of HD Agent Concentration. During the second 30 minutes of hot water spraying, the MINICAMS® monitoring indicated that the chamber air was saturated. Since the TC was visibly clean, it was necessary to take a DAAMS sample to determine the vapor level of agent concentration inside the TC. The TC was purged with "clean" air from the chamber's compressed air line. The interface plate and the 3 inch mounting plate used for the DAAMS sample were installed. The drain line was sealed with parafilm and the compressed air line was hooked up to one of the DAAMS ports. The other DAAMS port was used as an outlet. The five minute purge began at 19:11 and ended at 19:16 on 24 July. The compressed air line was then removed and the ports were covered with parafilm. The interior of the TC was then allowed to off-gas overnight. On 25 July 1996, four DAAMS tubes were installed: two for the EAI Sample Processing Area Laboratory at E3726 and two for the ERDEC Monitoring Team to analyze. The DAAMS sample was started at 09:20 and the pump sampled for two hours. The ERDEC Monitoring Team's DAAMS tubes had been calibrated with the pump that was used. Although the pump was set for 250 ml/min, the flow rates ranged from 215 to 225 ml/min. These actual flow rates were used for the calculation of agent concentration for the ERDEC Monitoring Team's DAAMS tubes. The EAI DAAMS tubes were not calibrated with the pump; and therefore, the set point of 250 ml/min was used in the calculations. EAI results for both DAAMS tubes indicated that the level of vapor concentration was less than 0.5 TWA; therefore, a 3X condition was reached. The ERDEC Monitoring Team results confirmed that a 3X condition was reached and reported an HD concentration of 6.7 nanograms, which correlated to 0.09 TWA. These results were confirmed by the mass spectrometer.

3.5.7.3 Additional Inspections. The weight of the 3X TC was 1554 pounds. A total of approximately 359 pounds of solids were removed from the TC during the demonstration. A total of 3.4 pounds of solids were collected during the initial characterizations and 20 grams were collected in the strainer; therefore, the remaining solids (approximately 355 pounds) went into aqueous solution that passed through the strainers and into the holding or decontamination tanks. A summary of the weights can be found in Table 3-11. Based on the inlet flow totalizers, a total of 315 gallons of water was used during the 10 minutes of eductor tube flushing and the 60 minutes of hot water spraying.

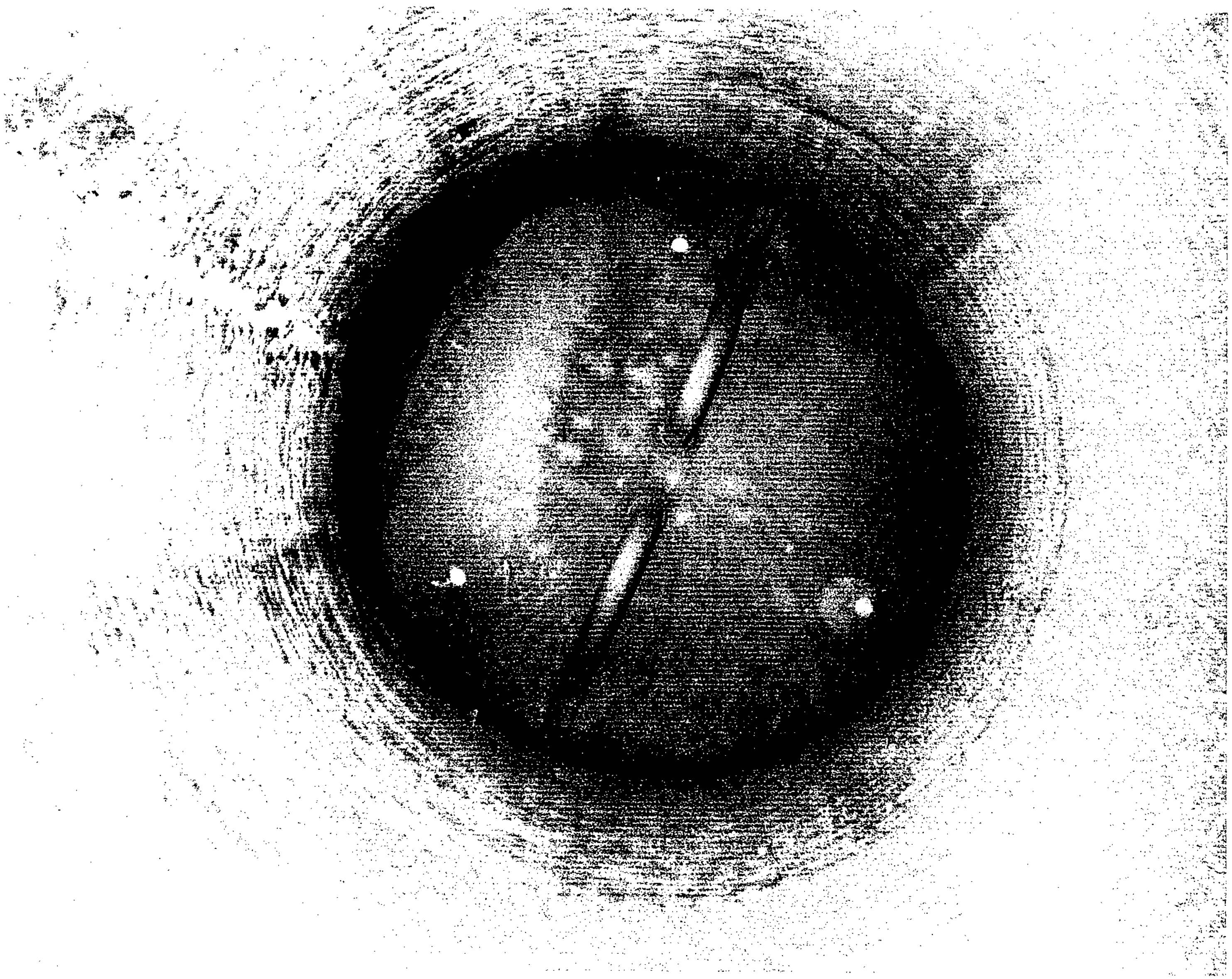


Figure 3-17. Interior of HD TC After 60 minutes of Hot Water Spraying

Table 3-11. Summary of Weights

Item	Weight (in pounds)
Initial TC Weight	1954
Items Removed from TC:	
Bonnet	12.68
Marking Plate	0.20
Ten Inch Disk	17.56
3 Plugs from Hole End	1.18
Shavings from Hole Cutting	1.82
3 Plugs from Valve End	1.22
2 Valves	6.18
Final Weight of TC	1554
Weight of Residue Removed	359.16

3.5.8 Post Cleaning Activities

Since the interior of the TC was now in a 3X condition, it was necessary to further decontaminate the chamber to bring the levels of HD agent concentration to below 1 TWA so that the entire TC could be "bagged" and monitored. All system lines were flushed with water. All equipment was cleaned and bagged and all possible sources of HD were eliminated; however, at times, the MINICAMS® was saturated. The MINICAMS® operator decided to swap two of the MINICAMS® - the one that monitored the chamber and the one that monitored the egress rooms. The MINICAMS® that had monitored the chamber was more sensitive to thiodiglycol interferences. After the swap on 29 July 96, the agent levels in the egress rooms remained at 0.0, while the agent levels in the chamber were approximately 6 to 7 TWA - this MINICAM® did not become saturated.

It was assumed that the root of the problem was still with the 1000 gallon holding tank and the 600 gallon decontamination tank, even though they were both cleaned a number of times with water and HTH bleach. Both tanks, including the lids, were then thoroughly scrubbed with decon a few more times, and this time, the concentration of HD agent inside the chamber fell to below 1 TWA.

The entire TC was then "bagged" and allowed to off-gas over the weekend of 9-11 August. A DAAMS vapor sample was taken on 12 Aug 96. Results from the ERDEC Monitoring Team indicated that the TC was in a 3X condition and was "clear of HD".

3.6 Analytical Results

The following paragraphs describe the analytical results. A summary of the analytical tests that was requested as part of the second HD Ton Container Cleanout Demonstration can be found in Tables 3-12 and 3-13. The methods that were used for each analysis are detailed in the HD Ton Container Survey and Ton Container Cleanout Analytical Test Plan (Reference 5).

Table 3-12. Summary of Analyses for Characterization Samples.

Sample Type	HD Agent	Metals	NMR	VOCs	Land bans	Flash Point	Purity
Neat Agent from TC Survey						x	
Neat Agent from TC		x			x		x
Solid Heel	x	x	x				
Vapor (initial & effluent)	x			x			
Water Benchmark		x					

Table 3-13. Summary of Analyses for Effluent Samples.

Sample Type	HD Agent	TDG	Metals	NMR	Organics	TSS	PSD
Liquid Effluent	x	x	x	x	x	x	
Solid Effluent	x		x	x			
Hold Tank Sample	x	x	x	x	x	x	
Settle Tank Sample	x	x	x	x	x	x	x

where NMR = Nuclear Magnetic Resonance

VOC = Volatile Organic Compounds

TSS = Total Suspended Solids

PSD = Particle Size Distribution

3.6.1 Characterization Samples

3.6.1.1 TC Neat Agent. Neat HD agent was analyzed for purity, flash point and "land ban" compounds by the Analytical Chemistry Team in Building E3300. "Land ban"

compounds are a specific set of organic compounds and metals identified by the Resource Conservation and Recovery Act (RCRA). A sample obtained from the pumpkin filled mid-way during the draining process of the TC selected for this demonstration was analyzed for purity, metals, and "land bans". Also, a TC survey sample was analyzed for flash point to confirm previous results.

3.6.1.1.1 Agent Purity. After draining the HD TC selected for this demonstration (TC #D94102) into pumpkins, a 10 milliliter sample (HD-S-6176-CTF-N) was taken from the middle pumpkin by the CTF. The purity of the liquid HD was determined by the Analytical Chemistry Team to be 92.9 +/- 0.1% from a gas chromatography/thermal conductivity detector (GC/TCD) characterization using ATP Method TC-03. This result was based on an average from three injections.

3.6.1.1.2 Organic Compounds. The neat agent sample taken from the mid-way pumpkin during the draining process was also characterized by gas chromatography/mass spectroscopy (GC/MS). This characterization resulted in an HD purity of 91.38%. An impurity, 1,2-dichloroethane, was reported as having an area percent of 0.35%. Compounds from a gas chromatography/mass spectroscopy/ electron ionization (GC/MS/EI) analysis and their resulting area percentages are listed in Table 3-14.

Table 3-14. Compounds in TC #D94102 Neat HD Agent

Compound	Area %
HD	91.38
Q	6.08
2-chloroethyl 4-chlorobutyl sulfide	0.86
1,4-dithiane	0.81
1,2-dichloroethane	0.35
Bis 3-chloropropyl sulfide	0.18
2-chloropropyl 3'-chloropropyl sulfide	0.18
2-chloroethyl 3-chloropropyl sulfide	0.14
1-chloropropyl 2-chloroethyl sulfide	0.02
1,4-thioxane	<0.01

The organic compounds for the neat agent in TC #D94102 were also documented in the Ton Container Decontamination and Disposal Program - Special Studies Report (Reference 1). These organic compounds are listed in Table 3-15.

Table 3-15. Compounds in TC#D94102 Neat HD (per Special Studies Report)

Compound	Area %
HD	89.2
Q, 1,2-bis-(2-chloroethylthio)ethane	4.7
Dichloroethane	2.4
Miscellaneous Isomers of $ClCH_2CH_2SCH_2CH_2CH_2CH_2Cl$	2.0
1,4-Dithiane ($C_4H_8S_2$)	1.2
Chloroethyl Chloropropyl Sulfide	0.4
Bis (2-chloroethyl) disulfide	0.1
1,4,5-Trithiepane ($C_4H_8S_3$)	0.1
Iron (as Fe^{+3})	7 atomic

3.6.1.1.3 Flash Point. Sample #HD-S-5179-CTF-N-1S from HD TC #D94041 (a TC Survey sample) was selected for flash point analysis since it was identified as having the "worst case land bans" as it related to flash point. It had the highest normalized weight percent (0.65%) of 1,2-dichloroethane according to a GC/TCD analysis. Of the "land ban" compounds found in the TC Survey samples, 1,2-dichloroethane had the lowest flash point (60°F). The flash point analysis was performed by the Analytical Chemistry Team using ASTM D3828-93 (Flash Point by Small Scale Closed Tester). Results indicated that this sample had a flash point of 102±1°C.

3.6.1.1.4 Metals. The neat agent sample from TC #D94102 (HD-S-6176-TC) was analyzed for metals using a Perkin Elmer Plasma II Inductively Coupled Plasma (ICP) spectrometer per ATP Method TC-05. ICP uses an argon torch at 5000°C to excite the metal ions in the sample. The emission lines are passed through an Eschel refractive grating and a photomultiplier is used to quantitate the presence of metals. The neat agent sample was also analyzed for mercury using the cold vapor technique; however, the sample matrix resulted in background levels rendering the cold vapor technique inappropriate. Due to unknown compounds in the matrix, cold vapor analysis was unable to reliably analyze this sample. The concentrations of various metals in the neat agent can be found in Table 3-16. As expected, there were high amounts of sulfur and iron present in the neat agent. Sulfur had a concentration of 264,420 parts per million (ppm), while iron had a concentration of 5,035 ppm.

Table 3-16. Concentration of Metals in Neat HD Agent (TC #D94102)

Metal	Content (ppm)
Aluminum	<13
Antimony	<9
Arsenic	18.9
Barium	<0.3
Beryllium	<0.2
Bismuth	<12
Cadmium	<3
Calcium	9.6
Chromium	<2
Cobalt	<2
Copper	91.8
Iron	5,035
Lead	<7
Magnesium	<0.4
Manganese	0.6
Nickel	<4
Phosphorus	32.6
Selenium	<14
Silicon	110.9
Silver	<4
Sodium	<16
Sulfur	264,420
Thallium	<13
Thorium	<11
Tin	<6
Vanadium	<3
Zinc	<0.7

3.6.1.2 TC Heel Characterization. Three "solid" samples of the TC heel were analyzed by Direct Exposure Probe (DEP)/MS and by Nuclear Magnetic Resonance (NMR) Spectroscopy. In addition, these samples were analyzed by inductively coupled plasma (ICP) and the cold vapor technique to determine the content of various metals and mercury.

3.6.1.2.1 Organic Compounds in TC Heel (by DEP). The three solid heel samples were analyzed by DEP/MS in the methane CI mode. Observed in all three spectra were HD, 1,4-dithiane, and Q; which suggests the solids were probably the sulfonium ion S-2-chloroethyl and 1,4-dithiane.

3.6.1.2.2 Complex Organic Compounds in TC Heel (by NMR). In order to determine the ratio of HD to the cyclic sulfonium ion, the samples were hydrolyzed for at least 48 hours in 1N HCl and analyzed by ^{1}H and ^{13}C NMR. The ^{1}H spectra were not used because of severe broadening of the resonance lines by paramagnetic ions in the solution. When hydrolyzed in 1N HCl, HD forms Thiodiglycol (TDG) and the sulfonium ions, CHTG and H_2TG, which are shown below:

CHTG	$HOCH_2CH_2SCH_2CH_2S{+}(CH_2CH_2OH)_2$
H_2TG	$(HOCH_2CH_2)_2S{+}CH_2CH_2SCH_2CH_2S{+}(CH_2CH_2OH)_2$
TDG	$HOCH_2CH_2SCH_2CH_2OH$

The results, as shown in Table 3-17, were reported as the mole ratio of the compounds.

Table 3-17. Complex Organic Compounds in HD Heel by NMR (mole ratio)

Sample #	Location	HD	Q Cyclic Sulfonium Ion*	1,4 Dithiane	Other
C-03-04-HH-1936	Top of peak	53	42	-	5
C-03-05-HH-1936	Bottom of heel	33	60	1	6
C-03-06-HH-1936	Middle portion	14	86	-	-

*where the Q cyclic sulfonium ion is as follows:

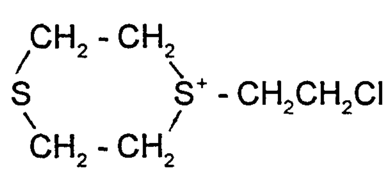


Table 3-18. Concentration of Metals in Solid Heel Samples (ppm)

Metal	C-03-04-HH-1936 (Top of Peak)	C-03-05-HH-1936 (Bottom of Heel)	C-03-06-HH-1936 (Middle Part)
Aluminum	43.1	<13	35.9
Antimony	<9	<9	<9
Arsenic	64.3	41.4	50.7
Barium	0.50	0.60	<0.3
Beryllium	<0.2	<0.2	<0.2
Bismuth	<12	<12	<12
Cadmium	14.6	12.1	14.7
Calcium	26.6	18.3	21.6
Chromium	34.7	34.7	42.8
Cobalt	14.3	10.8	11.4
Copper	129.5	72.5	67.6
Iron	125,813	93,011	108,292
Lead	14.3	11.7	15.9
Magnesium	77.4	6.5	60.9
Manganese	486.1	408.2	494.7
Mercury	0.63	0.15	0.95
Nickel	8.90	6.87	6.51
Phosphorus	35.3	22.4	10.0
Selenium	21.1	19.9	20.6
Silicon	94.6	20.6	56.1
Silver	<4	<4	<4
Sodium	115.0	26.4	23.7
Sulfur	244,168	233,108	239,629
Thallium	<13	17.1	17.4
Thorium	93.2	70.4	86.8
Tin	10.8	10.6	16.2
Vanadium	28.5	24.8	26.6
Zinc	226.5	145.7	185.3

3.6.1.2.3 Metals in TC Heel. The characterization samples were analyzed for metals, excluding mercury, using a Perkin Elmer Plasma II Inductively Coupled Plasma (ICP). The characterization samples were also analyzed for mercury using the cold vapor technique. As expected and seen in Table 3-18, extremely high amounts of iron and sulfur were observed. The iron content in the three characterization samples averaged approximately 109,000 parts per million (ppm), whereas, the sulfur content averaged approximately 240,000 ppm. More iron was found in the top of the peak sample than the bottom or middle samples. The sulfur content appeared to be relatively uniform for all three samples. The only other two metals that averaged greater than 100 ppm were manganese, which averaged 463 ppm, and zinc, which averaged 185 ppm. It should also be noted that the content of mercury ranged from 0.146 to 0.948 ppm.

3.6.1.3 TC Vapor Characterization. The initial vapor sample from the interior of the TC was taken with a SUMMA® canister and analyzed by the Analytical Chemistry Team. The SUMMA® canister was pressurized to 20 psi with nitrogen, and allowed to equilibrate for 16 hours. The canister was placed in a 60°C water bath for one hour. A 3 milliliter sample of the contents was taken and analyzed on a Hewlett Packard (HP) 5890 GC, which was interfaced to a cryofocusing trap maintained at minus 100°C. The trap was heated ballistically to 250°C and the trapped analytes were injected into a capillary column, which was interfaced to an HP 5970B Mass Spectrometer (MSD). The analytes (HD and Volatile Organic Compounds) were identified by mass spectrometry and are summarized in Table 3-19. In addition to HD, the main VOC was 1,2-dichloroethane, which had a concentration of 6.00 milligrams/liter (mg/L).

Table 3-19. Content of HD Agent and VOC's in Initial TC Vapor

Compound	Concentration (mg/L)
1,2-dichloroethane	6.00
Bis-(2-chloroethyl) sulfide (HD)	0.83
1,4-dithiane	0.6
2-Chlorobutane	0.21
Tetrachloroethene	0.083

3.6.2 Liquid and Solid Cleanout Effluent Samples

The initial characteristics, including the number of layers, the color, and the initial pH, of the liquid cleanout effluent samples as identified by the Analytical Chemistry Team can be found in Table 3-20. All samples, except sample #10, had one layer. For sample #10 (which was the first liquid effluent and also contained the highest concentration of

HD), two layers were present. The top layer, which was the larger of the two layers, was yellow in color. The bottom was a small dark layer. Analysis of the bottom layer revealed neat HD at a purity greater than 90%. The pH of the samples taken during the first 30 minutes of hot water spraying was below 1. After 60 minutes of hot water spraying, the pH had increased to between 5 and 6.

Table 3-20. Initial Characteristics of the Liquid Effluent Samples

Sample #	Time	Sample Type	# of Layers	Color	Initial pH
C-03-10-HH-1986	10	Liquid Effluent	2	top - yellow bottom - dark	0-1
C-03-13-HH-1986	20	Liquid Effluent	1	yellow	0-1
C-03-16-HH-1986	30	Liquid Effluent	1	lime green	0-1
C-03-19A-HH-1986	30	Hold Tank	1	lime green	0-1
C-03-F1-HH-1996	30	TC liquid	1	clear	0-1
C-03-F2-HH-1996	30	Strainer liquid	1	light green, solids on bot	0-1
C-03-20-HH-2066	45	Liquid Effluent	1	cloudy w/ sediment	4
C-03-23-HH-2066	60	Settle Tank Sample	1	cloudy w/ sediment	5
C-03-F5-HH-2066	60	Settle Tank Sample	1	clear w/ sediment	5-6
C-03-F6-HH-2086	60	Settle Tank Sample	1	clear w/ sediment	5-6

3.6.2.1 HD Agent. The HD content was determined by the Analytical Chemistry Team (ACT) using the parameters from ATP Method HN-01. For the first few liquid effluents (Samples #10, 13, 16, 19A, F2), the pH was extremely low and was adjusted before analysis to between 6 and 7. After adding NaOH, the samples appeared brown with many solids. Sample #F1 was also adjusted, however, the sample appeared green after adding the NaOH. Chloroform was used in the extraction and the extract was filtered before analysis. Samples were analyzed with an HP 5890 GC and a 5970 MSD. Results for the concentration of mustard in the liquid effluent samples can be found in Table 3-21 and Table 3-22. The concentration of HD was plotted against time

and can be found in Figure 3-18. The concentration of HD decreased to below 200 parts per billion (ppb) after 45 minutes of hot water spraying.

Table 3-21. HD and TDG Concentration for Effluent Samples

Sample #	Time	Sample Type	HD Content (ppm)	TDG Content (ppm)
C-03-10-HH-1986	10	Liquid Effluent	3,256	4,310
C-03-13-HH-1986	20	Liquid Effluent	2,302	10,370
C-03-16-HH-1986	30	Liquid Effluent	1.16	7,049
C-03-20-HH-2066	45	Liquid Effluent	< 0.2	442.7

3.6.2.2 Thiodiglycol. The thiodiglycol (TDG) concentration of the effluent samples was determined using ACT Method 030. A sample extraction was performed in accordance with ATP Method HN-02. The TDG analysis was performed on the aqueous phase remaining after the extraction. The concentration of TDG in the liquid effluent samples can be found in Table 3-21 and Table 3-22. The concentration of thiodiglycol was plotted against time and can be found in Figure 3-18. When the concentration of HD decreased to below 200 ppb, the TDG concentration was approximately 443 ppm. As expected, the HD concentration decreased over time; however, the TDG concentration initially increased to 10,370 ppm (after 20 minutes of hot water spraying) and then decreased. Keep in mind that the effluent pump was started at 10 minutes and a sample was taken after flow was observed; therefore the 10 minutes sample may not be a good representation of the effluent - it is just a point sample. It should also be noted that the cleaning was performed at a higher temperature which causes the HD reaction to move forward at an increased rate. This could also be the reason for the rise in TDG present in the effluent at 10 minutes.

Table 3-22. HD and TDG Concentration for Tank Samples & Special Samples

Sample #	Time	Sample Type	HD Content (ppm)	TDG Content (ppm)
C-03-19A-HH-1986	30	Hold Tank	3.4	11,950
C-03-F1-HH-1996	30	TC Liquid	0.07	Not requested
C-03-F2-HH-1996	30	Strainer Liquid	1.44	Not requested
C-03-23-HH-2066	60	Settle Tank	< 0.2	29.01
C-03-F5-HH-2066	60	Settle Tank	< 0.2	Not requested
C-03-F6-HH-2086	60	Settle Tank	< 0.2	Not requested

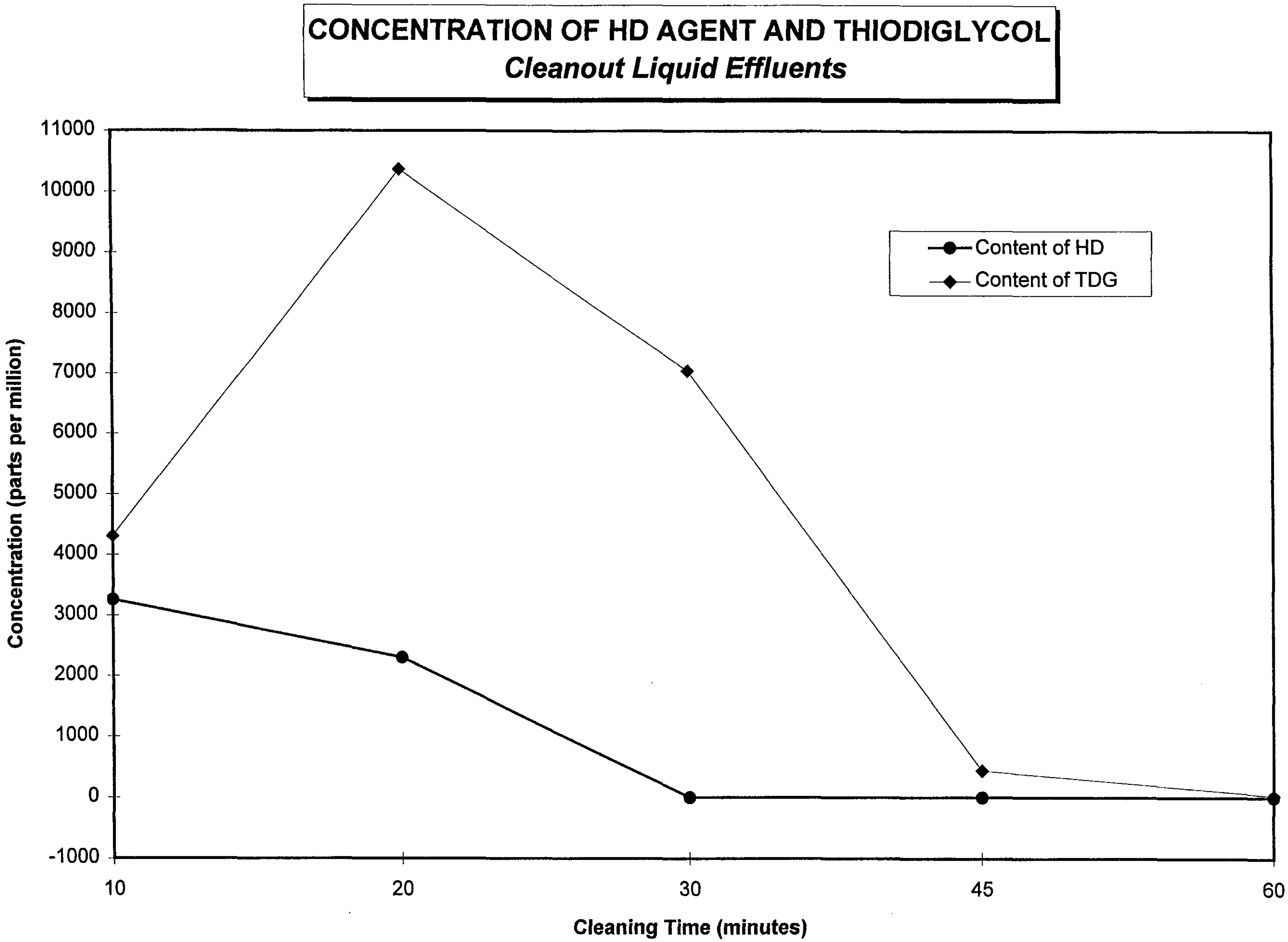


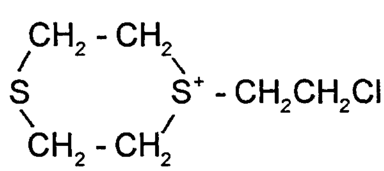
Figure 3-18. Concentration of HD Agent and Thiodiglycol in Liquid Effluent Samples (including settle tank sample at 60 minutes)

3.6.2.3 Complex Organic Compounds (by NMR). The liquid cleanout effluents, holding tank sample, and the settle tank samples were also analyzed by NMR. The samples were kept in the refrigerator at 4°C when not in the spectrometer. Both ^{1}H and ^{13}C NMR data were collected; however, the ^{1}H data was severely broadened by the presence of paramagnetic ions (presumably iron). Only the ^{13}C data, which was not as broad, was used to obtain the results, which can be found in Table 3-23. In these liquid effluent samples, the cyclic sulfonium ion was the most abundant organic compound.

Table 3-23. Complex Organic Compounds in Liquid Effluents and Hold Tank Samples from First 30 minutes of Hot Water Spraying (mole ratio)

Sample #	Type	Cyclic S+	TDG	H_2TG	1,4 Dithiane	Other
C-03-10-HH-1986	10 min	80	6	3	5	6
C-03-13-HH-1986	20 min	74	13	5	5	3
C-03-16-HH-1986	30 min	68*	26	2	1	3
C-03-19A-HH-1986	30 min (Hold Tank)	71	17	2	1	8

* Two cyclic sulfonium ions were present. The major cyclic sulfonium was present at 61 mole percent and is shown below. The structure of the second cyclic ion could not be elucidated; only the $S(CH_2CH_2)_2S+$ resonances were detected. The second ion may also be present in the other three samples, but the resonances in the spectra were too broad to resolve into individual ions. The major cyclic sulfonium ion is as follows:



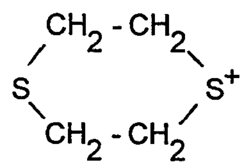
Both ^1H and ^{13}C NMR were used to characterize the 45 minute liquid effluent sample (C-03-20-HH-2066) and the results are shown in Table 3-24.

Table 3-24. Complex Organic Compounds in 45 min Liquid Effluent Sample (Sample C-03-20-HH-2066)

Compound	Mole Ratio of Compounds by ^1H NMR	Mole Ratio of Compounds by ^{13}C NMR
$R\text{-}CH_2CH_2Cl$	69	60
$R\text{-}CH_2CH_2OH$	Included Above	3
$R\text{-}CH{=}CH_2$	Included Above	6
TDG	22	21
H_2TG	Not Observed	1
1,4-Dithiane	8	6
Other	1	2

where,

R =



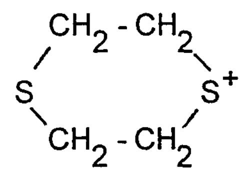
H_2TG = $(HOCH_2CH_2)_2S{+}CH_2CH_2SCH_2CH_2S{+}(CH_2CH_2OH)_2$
TDG = Thiodiglycol
Other = Includes the Q hydrolysis product, HO~ S~ S~ OH

Both ^1H and ^{13}C spectra were run for sample C-03-23-HH-2066, the settle tank sample after 60 minutes of hot water spraying. No resonances were detected in the ^{13}C spectrum after an overnight accumulation because the sample was too dilute. The ^1H NMR characterization of the sample is shown in Table 3-25.

Table 3-25. Complex Organic Compounds in Settle Tank Sample (C-03-23-HH-2066)

Compound	Mole Ratio of Compounds by ^{1}H NMR
$R\text{-}CH_2CH_2X$	4
$R\text{-}CH{=}CH_2$	29
TDG	36
Q-OH	7
1,4-Dithiane	18
Other	6

where,

R = 

X = OH or Cl
TDG = Thiodiglycol

A portion of the "wet" solid sample from the coarse strainer after 10 minutes of hot water spraying was dissolved in 1N HCL and allowed to hydrolyze for 24 hours before analysis by ^{13}C NMR. The results were reported as mole ratios and can be found in Table 3-26. Once again, the cyclic sulfonium ion comprised most (approximately 73%) of the sample. The HD was calculated from the TDG and the sulfonium ions (H_2TG and CHTG) formed on hydrolysis in the 1N HCl. The ^{13}C resonances were very broad indicating that this sample probably contained a lot of iron.

Table 3-26. Complex Organic Compounds in Strainer Solid Sample (mole ratio)

Sample #	Type	HD	Cyclic Sulfonium Ion	Other
C-03-11-HH-1986	Strainer - 10 min	25	73	2

3.6.2.4 Organic Compounds (by GC/FID). The organic composition of the liquid effluent samples was determined by the Analytical Chemistry Team using ATP Method HN-04. Identification was determined by GC/MS/EI and GC/MS/CI (gas chromatography/mass spectroscopy/chemical ionization). Thiodiglycol could not be quantitated because of HD interference. The compounds identified for each of the liquid effluent samples can be found in Tables 3-27 and 3-28. In all samples, 1,4-dithiane was the most abundant organic compound. These results differ from the NMR results since the sulfonium ion (both linear and cyclic) breaks down in the GC/MS to form 1,4-dithiane.

Table 3-27. Organic Composition of Effluent Samples (in mg/L)

Sample #	Time	1,4 Thioxane	/\S~OH	1, 4 Dithiane	/\S~S~OH	HO~S~S~OH
10	10	27.67	86.52	272.47	63.53	44.64
13	20	31.15	68.68	351.05	43.67	44.91
16	30	14.59	14.91	152.5	12.73	...
20	45	10.64	...	99.22	...	...

Table 3-28. Organic Composition of Tank Samples (in mg/L)

Sample #	Time	1,4 Thioxane	/\S~OH	1, 4 Dithiane	/\S~S~OH	HO~S~S~OH
19A (Hold Tank)	30	27.87	44.04	420.1	32.09	16.3
23 (Settle Tank)	60	...	...	16.3	...	....

3.6.2.5 Metals in Liquid and Solid Effluent Samples. The liquid and solid effluent samples were analyzed for metals using Inductively Coupled Plasma (ICP). The results can be found in Tables 3-29 and 3-30. For samples C-03-20-HH-2066 (45 minute sample) and C-03-23-HH-2066 (settle tank sample), the concentration of mercury was determined to be below 0.225 ppm using the cold vapor technique. For the remainder of the effluent samples, the concentration of mercury could not be determined. The determination of mercury for these samples was attempted using the cold vapor technique; however, the sample matrix resulted in background levels rendering the cold vapor technique inappropriate. Due to unknown compounds in the matrix, cold vapor analysis was unable to reliably analyze these samples.

Table 3-29. Content of Metals in Liquid and Solid Effluent Samples (ppm)

Metal	C-03-10... 10 min Liquid	C-03-11... 10 min Solid	C-03-13... 20 min Liquid	C-03-16... 30 min Liquid	C-03-20... 45 min Liquid
Aluminum	<1.95	<195	<1.95	<1.95	0.96
Antimony	<1.35	<135	<1.35	<1.35	<0.09
Arsenic	4.11	44.51	4.76	1.07	0.28
Barium	<0.045	<4.5	<0.045	<0.045	0.021
Beryllium	<0.03	<3	<0.03	<0.03	<0.002
Bismuth	<1.8	<180	<1.8	<1.8	<0.12
Cadmium	5.75	<45	5.15	<0.45	0.13
Calcium	<0.3	<30	<0.3	<0.3	1.76
Chromium	3.75	<30	3.70	<0.3	1.23
Cobalt	14.00	<30	17.85	32.45	0.14
Copper	43.45	<30	49.35	2,230	1.43
Iron	33,928	80,369	31,423	2,230	776.5
Lead	4.40	<105	4.75	286.3	0.10
Magnesium	<0.06	<6	0.50	<0.06	2.12
Manganese	141.3	393.9	129.5	8.00	5.04
Nickel	4.55	<60	6.55	2.90	1.75
Phosphorus	<1.5	<150	<1.5	<1.5	0.12
Selenium	<2.1	<210	<2.1	<2.1	<0.14
Silicon	<0.45	<45	<0.45	<0.45	5.32
Silver	<0.6	<60	49.35	<0.6	<0.04
Sodium	<2.4	<240	<2.4	808.5	26.64
Sulfur	74,068	152,026	66,028	6,002	1,801
Thallium	8.05	<195	<1.95	<1.95	<0.13
Thorium	22.00	<165	18.25	<1.65	0.84
Tin	<0.9	<90	<0.9	<0.9	<0.06
Vanadium	<0.45	<45	<0.45	<0.45	0.22
Zinc	53.5	<750	54.60	1,425	1.46

Table 3-30. Content of Metals in Tank and Special Samples (ppm)

Metal	C-03-19A... Hold Tank @ 30 min	C-03-23... Settle Tank @ 60 min	C-03-F1... TC Liquid @ 30 min	C-03-F2... Strainer Liquid @ 30 min
Aluminum	<1.95	0.51	<1/3	<1.3
Antimony	<1.35	<0.09	<0.9	<0.9
Arsenic	1.22	<0.05	<0.5	<0.5
Barium	<0.045	0.018	<0.03	<0.03
Beryllium	<0.03	<0.002	<0.02	<0.02
Bismuth	<1.8	<0.12	<1.2	<1.2
Cadmium	<0.45	<0.03	<0.3	<0.3
Calcium	<0.3	13.5	13.97	<0.2
Chromium	6.60	0.062	129.0	<0.2
Cobalt	1.45	0.025	<0.2	<0.2
Copper	11.55	0.46	46.83	<0.2
Iron	7,235	25.4	27,375	1,804
Lead	<1.05	<0.07	<0.7	<0.7
Magnesium	<0.06	4.28	7.03	6.92
Manganese	33.95	3.10	215.0	9.92
Nickel	3.95	0.094	83.23	<0.4
Phosphorus	<1.5	0.113	<1	<1
Selenium	<2.1	<0.14	<1.4	<1.4
Silicon	<0.45	1.00	71.10	<0.3
Silver	<0.6	<0.04	<0.4	<0.4
Sodium	<2.4	26.80	<1.6	<1.6
Sulfur	11,895	10,000	2,721,125	5,047
Thallium	<1.95	<0.13	<1.3	<1.3
Thorium	<1.65	<0.11	6,386	<1.1
Tin	<0.9	<0.06	<0.6	<0.6
Vanadium	<0.45	<0.03	13.733	<0.3
Zinc	0.827	0.16	62.70	5.25

As expected, the solid that was caught in the strainer after 10 minutes of hot water spraying (C-03-11-HH-1986) consisted of high concentrations of iron (80,369 ppm) and sulfur (152,026 ppm). The solid effluent also consisted of 394 ppm of manganese.

The primary metals that were found in the liquid effluents after 10 minutes, 20 minutes, 30 minutes and 45 minutes of hot water spraying were iron and sulfur, and as expected, the concentration of these metals decreased as more cleaning was performed. For the 30 minute sample (C-03-16-HH-1986), there was also a high concentration of copper, lead, sodium and zinc.

For the holding tank sample (C-03-19A-HH-1986), which was taken after 30 minutes of hot water spraying, the content of iron was 7,235 ppm while the content of sulfur was 11,895 ppm. The concentration of all of the other metals remained below 35 ppm. For the settle tank sample (C-03-23-HH-2066), which was taken after 60 minutes of hot water spraying, the concentration of iron had decreased to 25.4 ppm, while the conentration of sulfur had decreased to 10,000 ppm.

The special sample C-03-F1-HH-1996 was taken from the liquid inside the TC after 30 minutes of hot water spraying. At this stage, the liquid effluent was above 200 ppb and the vapor concentration was above 1 TWA. The concentration of sulfur was 2,721,125 ppm while the concentration of iron was 27,375 ppm. This sample also had high concentrations of copper, chromium, nickel, silicon, thorium and zinc.

The special sample C-03-F2-HH-1996 was taken from the liquid inside a strainer after 30 minutes of hot water spraying and after the holding tank recirculation was attempted. During the attempt at recirculating the holding tank, the effluent passed through this strainer. This sample contained high concentrations of iron (1,804 ppm) and sulfur (5,047 ppm).

3.6.2.6 Oxidation State of the Iron. The 45 minute liquid effluent sample (C-02-20-HH-2066) was analyzed to determine the total iron as well as the contents of ferrous (Fe^{+2}) and ferric (Fe^{+3}) iron. An Inductively Coupled Plasma (ICP) Spectrometer was used to determine the total amount of iron, while a Perkin-Elmer UV/VIS Spectrophotometer was used to determine the amount of ferrous iron in the sample. Ferric iron was determined by calculating their difference. The results can be found in Table 3-31.

Table 3-31. Content of Iron in 45 minute Liquid Effluent Sample

Total Iron	Ferrous (Fe^{+2})	Ferric (Fe^{+3})
2834 ppm	2444 ppm	390 ppm

3.6.2.7 Total Suspended Solids. The liquid effluent and tank samples listed in Table 3-28 were analyzed for total suspended solids by the ACT using ATP Method B-08. A measured amount of sample was filtered through a preweighed glass fiber filter disc. The filter disc was dried at between 103 and 105°C and the weight of the residue was compared with the volume of sample filtered. Results showed that there was approximately 270 to 280 ppm of suspended solids in these liquid effluent samples.

Table 3-32. Total Suspended Solids in Liquid Effluents

Sample #	Description	TSS (in ppm)
C-03-20-HH-2066	45 min	270
C-03-23-HH-2066	Settle Tank	279

3.6.2.8 Particle Size Distribution. The settle tank sample, C-03-23-HH-2066 (after 60 minutes of hot water spraying), was analyzed by the Environmental Technology Team for particle size distribution to assist in assessing whether or not the effluent at this stage (after solid heel removed from TC) could be recycled through the high pressure nozzle system. The diameters of the high pressure nozzles were also 0.007 inches. The laboratory received 105 ml (3.92 grams) of this sample and noted that it was cloudy with large particulate sediment. The sample was wet sieved using six stacked Tyler standard seives of the following sizes:

4 mesh	35 mesh
7 mesh	80 mesh
10 mesh	100 mesh
18 mesh	

Particles smaller than 100 mesh were collected on 1.2 micron membrane filters. The particles that were collected on the screens looked like iron oxide flakes. The results, which show the weight of the material on each screen per sample volume as well as the percentage of material (as compared to the total sample weight) that was found on each screen, can be found in Table 3-33.

Results indicated that approximately 57 weight (wt) percent of the particles collected ranged from 0.007 to 0.02 inches, 10 wt% ranged from 0.0059 to 0.007 inches, and 23 wt% were smaller than 0.006 inches. The remaining 10 wt% of the particles were greater than 0.02 inches. A summary of these ranges can be found in Table 3-34.

Table 3-33. Particle Size Distribution of Settle Tank Sample

Sieve #	Opening Diameter (microns)	Opening Diameter (inches)	Material Weight per 105 ml Sample (grams)	Percent of Total Weight
N/A	<149	<0.0059	0.9056	23.08
100	149	0.0059	0.3845	9.8
80	177	0.007	2.2502	57.35
35	500	0.0197	0.0731	1.86
18	1000	0.0394	0.1751	4.46
10	2000	0.0787	0.0874	2.23
7	2830	0.111	0.0471	1.2
4	5156	0.203	0.0	0.0

Table 3-34. Summary of Particle Size Distribution Results

Range of Particles (inches)	Weight Percent
< 0.0059	23.08
0.0059 - 0.007	9.8
0.007 - 0.02	57.35
> 0.02	9.75

3.6.3 Vapor Cleanout Effluent Samples

Prior to the demonstration, a benchmark vapor sample (C-03-02-HH-1926) was drawn from the 1000 gallon hold tank via a SUMMA® canister. Three 5 milliliter aliquouts of the contents of the SUMMA® canister were cryofocused and then injected into the GC/MS. The detection level for HD was 1 μg/ml. No HD or Volatile Organic Compounds (VOC's) were detected at or above the detection level. As stated in the previous section, it was planned that the valves to the 100 ml/min and 34 ml/min SUMMA® canisters were to be opened once effluent started flowing to the 1000 gallon holding tank. However, once the valves were opened, a vacuum was not observed - the canisters were already full. It was decided to send the SUMMA® canisters for

analysis even though the samples (C-03-21-HH-1986 and C-03-35-HH-1986) did not represent the vapor of the holding tank during the cleanout; therefore, these results are not representative of the vapors from the cleanout process. Due to leaks, it appeared that both canisters were filled after the hole was cut out of the TC but before the cleaning process began. The content of HD and the VOC's were determined. The concentration of HD was below the detection limit of 1 μg/ml. The volatile organic compounds found in each sample are listed in Table 3-35.

Table 3-35. Volatile Organic Compounds in Effluent SUMMA® Canisters (ng/ml)

VOC	C-03-21-HH-1986	C-03-35-HH-1986
1,2-dichloroethane	2	2
2,3-dihydrothiophene	1.0	1.6
1,4-oxathiane	1.0	1.7
1,4-dithiane	10.0	10.8
Chloroform	...	0.2

3.6.4 TC Vapor Contamination

A DAAMS vapor sample was taken after each stage of hot water spraying to determine if the interior of the TC was in a 3X condition. As explained in paragraph 3.5.7.2, both the EAI Sample Processing Area Laboratory at E3726 and the ERDEC Monitoring Team performed the analyses. A summary of the contamination levels of HD agent vapor can be found in Table 3-36.

Table 3-36. TC Vapor Contamination Results

Sample #	Description	EAI Results	Monitoring Results
C-03-F3-HH-2066	interior - 30 min	4.8 TWA	Not Analyzed
C-03-24-HH-2076	interior - 60 min	<0.5 TWA	0.09 TWA
0812001 (E3566 #)	entire TC	Not Analyzed	< 1 TWA

SECTION 4
SUMMARY OF TEST RESULTS

4. SUMMARY OF TEST RESULTS

4.1 Introduction

One of the objectives of the second HD Ton Container Cleanout Demonstration was to confirm the results of the first demonstration, mainly to confirm the composition of the heel, and the effectiveness of steam to flush out the eductor tubes and the effectiveness of pressurized hot water to remove the solids and to decontaminate the TC to a 3X condition (vapor space HD concentration less than 0.003 mg/m^3). In addition to providing a summary of the test results from the second HD TC Cleanout Demonstration, this section also compares these results to the results of the first HD TC Cleanout Demonstration.

4.2 Summary of Test Results from the Second HD TC Cleanout Demonstration

Objective a (as described in paragraph 3.1) was to confirm the ton container heel composition and mass. For the second HD TC (#D94102), approximately 359 pounds of heel was removed, whereas, for the first HD TC (#D93734), which was determined by NDE to be the "worst case", approximately 220 pounds were removed (Table 4-1). According to analyses from both demonstrations, the TC HD heel was primarily composed of three components: iron, HD, and the cyclic Q sulfonium ion.

Table 4-1. Comparison of Weights of HD Heel

TC #D93734 (First Demonstration)	TC #D94102 (Second Demonstration)
220 pounds	359 pounds

Objectives b and c (as described in paragraph 3.1) were to confirm the effectiveness of flushing the eductor tubes with steam prior to hot water spraying the interior of the TC to remove solids and residue inside the eductor tubes; and to confirm the effectiveness of a high pressure impingement hot water spray to remove solids and scale from the inside walls to achieve visibly clean surfaces and to achieve a 3X condition.

The first HD TC Cleanout Demonstration showed that a combination of steam and pressurized hot water was successful in removing the HD heel and decontaminating the "worst case" TC to a 3X condition. However, for this demonstration, problems with the hot water generators were encountered and after 30 minutes of hot water spraying, a

low flow rate of water and varying temperatures were observed. The demonstration continued since its main objective was to demonstrate that pressurized hot water could decontaminate a TC to a 3X condition. Results indicated that a 3X condition was reached; however, after a total of 6 hours of hot water spraying. Also, after a 3X condition was reached, residue in the eductor tubes was observed and the eductor tubes had to be flushed with hot water and steam to, once again, attain a 3X condition.

For the second HD TC Cleanout Demonstration, the eductor tubes were flushed out with steam (for 10 minutes) prior to spraying the interior of the TC with pressurized hot water. After 30 minutes of pressurized hot water spraying, all of the solid heel was removed and after 60 minutes of pressurized hot water spraying, the interior of the TC was determined to be in a 3X condition. After cleaning the chamber and equipment to decrease agent concentration levels in the chamber, the entire TC was determined to be in a 3X condition. Therefore, it was determined that flushing the eductor tubes prior to hot water spraying the interior was more efficient.

Objectives d and e (as described in Paragraph 3.1) were to determine the minimum quantity of water and time required to remove the heel and to achieve a 3X condition. It took approximately 10 minutes of eductor tube flushing with steam and 30 minutes of pressurized hot water spraying (and a total of 162 gallons of water) to remove the heel and achieve a visibly clean surface. An additional 30 minutes and 153 gallons of water were required to achieve a 3X condition; therefore a total of 70 minutes and 315 gallons of water was used to clean and decontaminate the HD TC #D94102 to a 3X condition.

For the cleanout and decontamination of the first HD TC, 255 gallons of water and 6 hours was required to attain a 3X condition. However, after a 3X condition was reached, HD residue that had crusted over was found in the eductor tubes. Once the crust was broken through for an agent test, the 3X condition was compromised. In order to once again, achieve 3X, the eductor tubes were flushed with steam for 30 minutes. Also, the inside of the TC had to be rinsed with pressurized hot water for 30 minutes and rinsed with steam for 85 minutes before a 3X condition was attained. During this time, approximately 300 gallons of water were used. A comparison of what was required to remove the heel and attain a 3X condition between the two demonstrations is shown in Table 4-2.

Objective f (as described in Paragraph 3.1) was to assess the feasibility of using recycled water through the high pressure systems after the TC was visibly clean (heel removed). For this demonstration, the heel was removed after 10 minutes of steaming the eductor tubes and 30 minutes of hot water spraying the inside of the TC. The effluent was not actually recycled as part of this demonstration. Recycling feasibility was assessed by measuring particle size distribution, fluid pH, organic composition, and particle composition in the effluent stream during the subsequent spray washes. Particle size distribution was determined by installing a settle tank in the effluent line. The settle tank was sized to capture any particles generated in the subsequent spray wash cycles that were larger than .007 inches in diameter. The TC spray effluent was

Table 4-2. Time and Water Required to Remove Heel and Achieve 3X

Condition	TC #D93734 (First HD TC)	TC #D94102 (Second HD TC)
Time Required to Remove Heel (Water Required)	30 minutes of hot water spraying (150 gallons)	10 min of steam flushing eductors & 30 min of hot water spraying (162 gallons)
Time Required to 3X (Water Required)	360 min hot water spraying, 30 min steam flushing eductors, & 115 min water/steam rinse (555 gallons of water)	10 min of steam flushing eductors & 60 min of hot water spraying (315 gallons)

directed to the upstream side of the settle tank weir for the entire spray cycle (ie, 30 minutes). At the conclusion of the wash cycle, a sample of the settle tank sediment was taken for analysis. The sediment therefore represents an accumulation of particle sizes generated during the spray cycle. The analysis of the accumulation determined the spectrum of particle sizes that will be expected to exist in the effluent stream and need to be removed if the effluent is to be recycled. Results of the analysis, as shown in Table 4-3, indicated that approximately 57 weight (wt) percent of the particles collected ranged from 0.007 to 0.02 inches in diameter, 10 wt% ranged from 0.0059 to 0.007 inches, and 23 wt% of the particles were smaller than 0.0059. The remaining 10 wt% were greater than 0.02 inches in diameter.

Table 4-3. Summary of Particle Size Distribution in Settle Tank

Range of Particles (inches)	Approximate Weight Percent
< 0.0059	23
0.0059 - 0.007	10
0.007 - 0.02	57
> 0.02	10

A total suspended solids analysis indicated that there were 270 to 280 ppm of suspended solids in the effluent during this stage of the demonstration. The pH at the beginning of this stage of cleaning, was 4 to 5, while at the conclusion of this stage, the pH was in the range of 5 to 6. The primary organic compounds included the cyclic sulfonium ion, TDG, and 1,4-dithiane.

Objective g (as described in Paragraph 3.1) was to quantify and characterize all solid, liquid, and vapor effluents generated during the pressurized hot water cleaning of the TC. This was accomplished for the second TC cleanout with one exception - the effluent vapor samples could not be collected due to equipment failure. These results from the second demonstration were also compared to the results from the first demonstration, as seen in the following paragraphs.

The second HD Ton Container Cleanout was a success as all objectives were met. The TC was cleaned and decontaminated to a 3X condition using only pressurized hot water and steam.

4.3 Comparison of Test Results

The results from the second HD TC (Serial #D94102) are compared to the results from the first HD TC (Serial #D93734) in the following paragraphs. Results from the first HD TC Cleanout Demonstration can be found in the Ton Container Decontamination and Disposal Program Demonstration Report: Mustard Agent HD Ton Container (Reference 7). Although similarities and differences, where applicable, are discussed; any similarities between the two TCs can not be made into generalizations or conclusions since it would be required to clean and analyze a specific number of HD TCs (based on a statistical approach) to obtain valid generalizations and conclusions regarding HD ton containers. Based on the results of two HD TC Cleanout Demonstrations, there are a number of similarities as well as differences between the two HD TCs. These differences range from physical dimensions and degree of difficulty in removing plugs and valves to differences in the characteristics of the heels and effluents from the cleanout process.

4.3.1 Neat Agent Characterization

The two results reported for the purity of the neat agent (based on two different analytical procedures) in TC #D94102 (TC Demo #2), 92.9% and 91.38%, coincides with the results from the Bulk Agent Stockpile Survey (Reference 4), which states that the average purity in the 25 randomly selected TCs was 91.49% with a standard deviation of 2.33%.

4.3.2 Heel Characterization

The heel from TC #D93734 (TC Demo #1) had both liquid HD and solids present; whereas, the heel from TC #D94102 (TC Demo #2) had no observable liquid since more efficient pumps were used to drain the TC. It has been shown that the HD heels from both TCs are mainly comprised of three components: iron salts, HD agent, and the

cyclic Q sulfonium ion. According to the metals and mercury analyses, both TCs primarily contained iron and sulfur, as expected. Other metals that were somewhat abundant (greater than 100 ppm) in the TC #D93734 (TC Demo #1) heel included copper, manganese, and zinc; while zinc and manganese were the only other metals that averaged greater than 100 ppm in the second HD TC. It should also be noted that there were distinct differences in the content of mercury. For the TC #D93734 (TC Demo #2) heel, the mercury content ranged from approximately 5.5 ppm to 14 ppm; whereas, for the second HD TC heel, the mercury content for 3 heel samples ranged from 0.15 to 0.95 ppm, as shown in Table 4-4.

Table 4-4. Concentration of Mercury in HD Heels

TC #D93734 (First HD TC)	TC #94102 (Second HD TC)
5.5 - 14 ppm	0.15 - 0.95 ppm

4.3.3 Vapor Characterization

The analysis of the vapor sample taken from the head space of the HD TCs prior to cleaning from both HD TCs showed different results. The major compounds found in the first HD TC included HD, 1,4-dithiane, and vinyl chloride. The major compounds found in the second HD TC included HD, 1,2-dichloroethane and 1,4-dithiane.

4.3.4 Liquid Effluents

For the first few liquid effluent samples for both demonstrations, the content of HD differed by approximately a magnitude of ten. For the first HD TC cleanout, the HD concentration for the 15 and 30 minute effluent samples were 22,080 ppm and 13,440 ppm, respectively. The HD concentration for the 10, 20, and 30 minute effluent samples were 3,256 ppm, 2,302 ppm, and 1.16 ppm, respectively. A summary of this comparison is shown in Table 4-5. Since the analytical data for the concentration of HD and TDG varied throughout the cleanout samples, it is difficult to note any comparisons or trends. For both cleanout demonstrations, the organic composition of the liquid effluents included the cyclic sulfonium ion, TDG, and 1,4-dithiane.

Table 4-5. Comparison of HD Concentration in Liquid Effluents (ppm)

Sample Time	TC #D93734 (First HD TC)	TC #D94102 (Second HD TC)
10 min	N/A	3,256
15 min	22,080	N/A
20 min	N/A	2,302
30 min	13,440	1.16

4.3.5 Solid Effluents

There were less solids collected in the strainers during the second demonstration, however, the organic composition of the solids was similar. Twenty grams were collected in the strainers during the second demonstration, while 0.69 pounds (313 grams) were collected during the first demonstration. The content of the cyclic sulfonium ion was 71-74%, while the content of HD was approximately 25-27% for all strainer samples analyzed.

4.3.6 Vapor Effluents

It was not possible to compare the volatile organic compounds in the effluent vapor since a representative sample of the vapor effluent was not obtained during the second demonstration due to equipment failure.

SECTION 5
CONCLUSIONS

5. CONCLUSIONS

The cleanout and decontamination of a second HD Ton Container (Serial #D94102) using only steam and pressurized hot water was successfully demonstrated. Steam was first used to flush out the eductor tubes, and then, pressurized hot water was used to remove the heel and to achieve a 3X condition (vapor concentration less than 0.003 mg/m^3). As stated in the TEMP, a vapor HD concentration level of less than 0.003 mg/m^3 was the only key criteria listed for Ton Container Cleanout.

The first HD TC Cleanout Demonstration indicated that steam and hot water were capable of decontaminating an HD TC to a 3X condition, and the results of the second demonstration confirmed this indication.

For the HD TC used in the second demonstration, the HD heel was removed (and the surfaces were visibly clean) after 10 minutes of flushing out the eductor tubes with steam and 30 minutes of hot water spraying of the TC interior. A total of 162 gallons of water was used to remove the heel. In order to achieve a 3X condition, 10 minutes of flushing the eductor tubes with steam and 60 minutes of hot water spraying of the interior of the TC were required. A total of 315 gallons of water was used to achieve a 3X condition.

Data was also obtained to assess the feasibility of recycling the effluent once the heel was removed. Results indicated that recycling this effluent is feasible.

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SECTION 6
RECOMMENDATIONS AND LESSONS LEARNED

6. RECOMMENDATIONS AND LESSONS LEARNED

6.1 Recommendations

The cleanout and decontamination of a second HD TC containing approximately 359 pounds of residue or heel was successfully demonstrated using a combination of steam (to flush out the eductor tubes) and pressurized hot water (to clean the interior of the TC). It is therefore recommended that flushing the eductor tubes with steam and then spraying the interior of the TC with pressurized hot water be used in the pilot plant design to cleanout and decontaminate HD TCs.

It has been observed that it is critical to spray closer to the TC end walls to thoroughly clean the TC. If the effluent from the second stage of cleaning (after the heel is removed) is to be recycled, it is recommended that appropriate strainers be placed in line to protect the pump and to keep the spray nozzle from clogging.

6.2 Lessons Learned

A number of findings were identified during the preparation and the execution of the demonstration that could have an impact on future demonstrations and/or on the design of a TC cleanout and decontamination process for the HD neutralization plant.

a. Although the NDE results from the Ton Container Survey were used to select the "worst case" TC and to determine whether or not TC #D94102 was to be used for the second demonstration, the NDE results appear to be inaccurate. For these two TCs, the NDE results were higher than what was actually observed. Some heel peaks or heel on a side wall may have interfered with the NDE results.

b. The TC valves and plugs, which need to be removed early in the cleaning process, may be difficult to remove either manually or automatically. For this demonstration, an 18 inch pipe wrench and a two-foot breaker bar were used to loosen the plugs and valves. It appears that the level of difficulty may be different for each TC.

c. If SUMMA® canisters are used in additional demonstrations and/or in the plant to collect vapor samples, it is necessary to evacuate the canisters prior to the sample being taken in case minor leaks are present. A leak or a leak rate test

should be performed on the SUMMA® canisters to determine if leakage is present. It is possible that any leakage, even minimal leakage, can affect sampling results.

d. For accurate levels of agent vapor contamination from a DAAMS, it is necessary to measure the flow rate through the sample tube before and after a sample is taken, and to use this flow rate (and not the pump set flow rate) in the calculations. Also, a mass flow controller could be placed in line to address this problem.

e. In all three demonstrations (two HD TCs and one VX TC), it has been shown that it is critical to spray closer to the plug and eductor walls to thoroughly clean the TC ends.

f. Solids will be caught in the strainers and adequate strainers should be used to protect equipment such as the pumps and the spray nozzles. For the pilot plant design, the disposal of the solids that are caught in the strainers will need to be addressed.

g. In all three demonstrations (two HD TCs and one VX TC), it was difficult to obtain a leak-free seal with the interface plate even with two o-rings installed. It appeared that the contour of each TC was slightly different and/or the interface plate had warped. Although it is currently planned in the plant design that a ten inch hole will not be cut out of a TC end (but two holes will be punched out on top of the TC), this equipment problem needs to be addressed. In the current plant design, there will be cover plates over these holes similar to the interface plate. These plates will be needed to prevent any splashback and to hold inlet and effluent lines in place. In addition, these plates will need to mate closely with the TC to minimize any leaks. In this demonstration, it was also observed that after some pressurized hot water spraying, the rubber o-ring seal toward the left side of the interface plate (which initially appeared sealed) failed and steam was seen escaping out of the TC (in addition to a liquid leak on the bottom of the interface plate). The high temperature and highly acidic solution may have been a factor in this situation. A rubber o-ring may not be an adequate seal for the interface plate. A different type of o-ring or a thick seal may be a better interface between the plate and the TC.

h. The effluent from the initial stage of hot water spraying (to remove the heel) was highly acidic with the pH less than 1. All equipment that will carry or store this effluent needs to be able to handle not only a high temperature solution but a highly acidic one as well.

i. A build-up of corrosion was seen on the wand after two demonstrations (a total of 8 to 10 hours of operation). This prevented the operators from manually moving the wand to its next position during the test. However, upon removing

the wand from the TC and partially disassembling the wand mounting fixture, operators were successful in moving the wand to its proper position. If the wand's position needs to be adjusted or moved relative to any mounting or shielding plate, then maintenance may be a concern. The wand may need to be coated or manufactured with materials that will resist corrosion. The wand may also need to be cleaned or rinsed after each use to stop any reactant attack that may be occurring while the wand is not in use.

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5. PMCD, HD Ton Container Survey and Ton Container Cleanout Analytical Test Plan (17 April 1996).

6. Test and Evaluation Master Plan for the Chemical Stockpile Disposal Program Alternative Technology Program. (May 1996).

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8. ERDEC, Ton Container Cleanout Standing Operating Procedure. SOP #CR93SP009 (4 March 1996).

9. ERDEC, E3566 Log Book #D1: Ton Container Cleanout Demonstrations. Pages 154-184. (1996).

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11. ERDEC, E3566 Second HD Ton Container Cleanout Demonstration Data Binder (July 1996).

12. PMCD, Ton Container Decontamination and Disposal Program Demonstration Report: A Second Mustard Ton Container (TC #D94102) - Volume 2 (20 November 1996).

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APPENDIX A
ACRONYMS/ABBREVIATIONS

ACAMS	Automatic Continuous Air Monitoring System
APCI	Atmospheric Pressure Chemical Ionization
APG	Aberdeen Proving Ground
AR	Army Regulation
ASTM	American Society for Testing and Materials
ATP	Alternative Technology Program
CASY	Chemical Agent Storage Yard
CI	Chemical Ionization
CSDP	Chemical Stockpile Disposal Program
CTF	Chemical Transfer Facility
DA	Department of the Army
DAAMS	Depot Area Air Monitoring System
DEP	Direct Exposure Probe
DET	Demonstration Evaluation Team
EA	Edgewood Area
EI	Electron Ionization
EPDM	Ethylenepropylene Dimonomer
ERDEC	Edgewood, Research, Development, and Engineering Center
FTIR	Fourier Transform Infrared Spectroscopy
GC	Gas Chromatograph
GPM	Gallons Per Minute
HD	Distilled Mustard Agent, bis (2-chloroethyl) sulfide
HP	Hewlett Packard
HTH	High Test Hypochlorite
ICP	Inductively Coupled Plasma
mg/L	milligrams per liter
mg/min	milligrams per minute
mg/m^3	milligrams per cubic meter
MINICAMS	Miniature Continuous Air Monitoring System
MPF	Metal Parts Furnace
MS	Mass Spectroscopy

NDE	Nondestructive Evaluation
NECD	Newport Chemical Depot
ng/ml	nanograms per milliliter
NMR	Nuclear Magnetic Resonance
NPT	National Pipe Thread
OIPT	Overarching Integration Product Team
PL	Public Law
PMAT&A	Product Manager for Alternative Technologies and Approaches
PMCD	Product Manager for Chemical Demilitarization
ppb	Parts Per Billion
PPE	Personal Protective Equipment
ppm	Parts Per Million
RCRA	Resource Conservation and Recovery Act
R&D	Research & Development
REC	Record of Environmental Consideration
SAIC	Science Applications International Corporation
SOP	Standard Operating Procedure
SS	Stainless Steel
SWEC	Stone & Webster Engineering Corporation
TC	Ton Container
TCD	Thermal Conductivity Detector
TDG	Thiodiglycol
TEMP	Test and Evaluation Master Plan
TSS	Total Suspended Solids
TTC	Toxic Test Chamber
TWA	Time Weighted Average
μg/ml	micrograms per milliliter
VOC	Volatile Organic Compounds
VX	Nerve Agent, O-ethyl S-(2-diisopropylaminoethyl) methylphosphonothioate

APPENDIX B
DEFINITIONS

3X Condition	A vapor space HD concentration less than 0.003 mg/m^3 when measured in a closed container (space) with ACAMS®, MINICAMS®, or DAAMS, as defined by AR 385-61. The HD vapor concentration of 0.003 mg/m^3 is also defined as 1 time-weighted-average (TWA).
Heel	The residue that cannot be pumped or drained out of the ton container.
"Land ban" compounds	A specific set of organic compounds and metals identified by the Resource Conservation and Recovery Act (RCRA).
Soak period	The initial 10 minutes of the hot water spraying process, in which, the water was allowed to fill the TC. The effluent pump was started at 10 minutes.
SUMMA® canister	A six liter evacuated sphere designed to collect a fixed volume of gases or vapors.
"Y Wand" hookup	The equipment used for steam flushing the eductor tubes; which consisted of EPDM hoses, fittings, and two valves (See Figure 3-7)

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APPENDIX C
DEMONSTRATION CHANGE FORM

Note: Enclosures in Appendix C have been re-numbered to coincide with current report.

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DEMONSTRATION CHANGE FORM

To:	PMAT&A	
From:	Darren W. Dalton, SCBRD-END	
Date:	3 July 96	
Test Plan:	HD Ton Container (TC) Cleanout Demonstration	**Subtest:** 3

Evaluation:
The 2nd HD Ton Container Cleanout Demonstration will be performed to determine the quantity of water and time required to remove the solid residue/heel from the ton container, to determine the quantity and time required to achieve a 3X condition using pressurized hot water, and to confirm the results of the 1st HD TC Cleanout Demonstration. The residue inside the TC and all solid and liquid effluents will be quantified and characterized. This subtest will follow all requirements as in Subtest 1, but with the following changes:

Changes:
1. (Page 1-2, Paragraph 1.1 Objective). Replace the objectives with the following:

"The specific objectives of this demonstration are the following:

a. Confirm ton container heel composition and mass by quantifying and characterizing residue remaining in the HD TC immediately following TC draining.

b. Confirm the effectiveness of flushing the eductor tubes with steam, prior to hot water spraying the interior of the TC, to remove solids and residue inside the eductor tubes.

c. Confirm the effectiveness of a high (up to 3000 psi) pressure impingement water spray using hot (90 +/- 5 deg C) water to remove solids and scale from the inside walls of an HD TC to achieve visibly clean surfaces.

d. Determine the minimum quantity of water and time required to remove the bulk of the heel material for a visually clean TC.

e. Determine the minimum quantity of water and time required to clean the TC to 3X.

f. Assess the feasibility of using recycled water through the high pressure system after the TC is visibly clean by measuring:

- particle distribution >.007" in the recycle stream
- fluid pH in the recycle stream
- fluid organic composition
- particle composition.

g. Quantify and characterize all solid and liquid effluents from pressurized hot water cleaning."

2. (Page 1-4, Paragraph 1.2 Concept). Replace the fourth paragraph (The demonstration will be performed ...) with the following:

To:	PMAT&A	
From:	Darren W. Dalton, SCBRD-END	
Date:	3 July 1996	
Test Plan:	HD Ton Container (TC) Cleanout Demonstration	**Subtest:** 3

"The demonstration will be performed using an HD TC drained at the CTF. This HD TC (Serial # 94102) was selected since the non-destructive evaluation results indicated that the heel was approximately 11 inches. Also, it was more cost and time effective to use a TC that was stored at the CTF, as opposed to one that was in the Chemical Agent Storage Yard".

3. (Page 1-5, Figure 1-2). Replace the As Built Drawing with the enclosed As Built Drawing (see Enclosure 1). The changes are as follows:
- The TC will be vented through one of the plugs on the eductor tube end during the flushing of the eductors and the initial "soak period" during the pressurized hot water spraying.
- There will be no methanol impinger sampler.
- The fine duplex strainer will be 80 mesh not 40 mesh.
- A settle tank and an additional pump will be installed following the coarse strainer and prior to the existing air diaphragm pump. The settle tank is 2' in diameter and 2' 3" in length. A weir is located 1' from the inlet. In addition to inlet and outlet lines, there will be a vent and a sample drain line.

4. (Page 2-3, Paragraph 2.5 Demo Procedure). Replace Paragraph 2.5 (with the exception of the last four paragraphs relating to analytical) with the attached demonstration procedures for the 2nd HD TC Cleanout (see Enclosure 2).

5. (Page 2-5, Paragraph 2.5 Demo Procedure). Samples will be numbered C-03-nn-HH-yyyz. A revised sample listing is included in Enclosure 2. Methanol impinger samples will not be collected since it has been determined that the vapor sample collected in the SUMMA canisters is sufficient. A tap water sample will be collected prior to the start of the demonstration. A one liter sample from the hold tank after 30 minutes of hot water spraying will be collected for Dr. Steve Harvey. This sample will be reacted and used for iron analysis. A five gallon sample of the hold tank will also be collected at this time.

6. (Page 2-8, Paragraph 2.8 Data Analysis Methods and Procedures). Replace Paragraph 2.8 with the Data Analysis Methods and Procedures for the 2nd HD TC Cleanout (see Enclosure 3).

7. (Page B-3 and throughout plan). Since SAIC has limited responsibilities with the 2nd HD TC Cleanout Demonstration, the additional responsibilities of the ERDEC Principal Investigator include prepares, coordinates, reviews and ensures written approval of all proposed changes to

To:	PMAT&A	
From:	Darren W. Dalton, SCBRD-END	
Date:	3 July 1996	
Test Plan:	HD Ton Container (TC) Cleanout Demonstration	**Subtest:** 3

the demo plan; and leads the preparation of the final demo report for the 2nd HD TCC Demo (which will be an addendum to the 1st report written by SAIC). SAIC will attend meetings from an integration standpoint, facilitate SWEC and ERDEC interaction, and ensure the addendum report be published and distributed as required in the Statement of Work.

8. (Appendix D: Data Collection Sheets). Replace the Data Sheets with the Data Sheets for the 2nd HD Ton Container Cleanout (see Enclosure 4).

Concurrence: Your concurrence with these changes is requested. Please sign and return.

Ltc Landry for Donald J. Palughi
Product Manager for Alternative Technologies & Approaches

8 Jul 96
Date

[signature]
Test Director, PMAT&A

8 Jul 96
Date

Joseph J. Novad
Team Leader, ERDEC

8 Jul 96
Date

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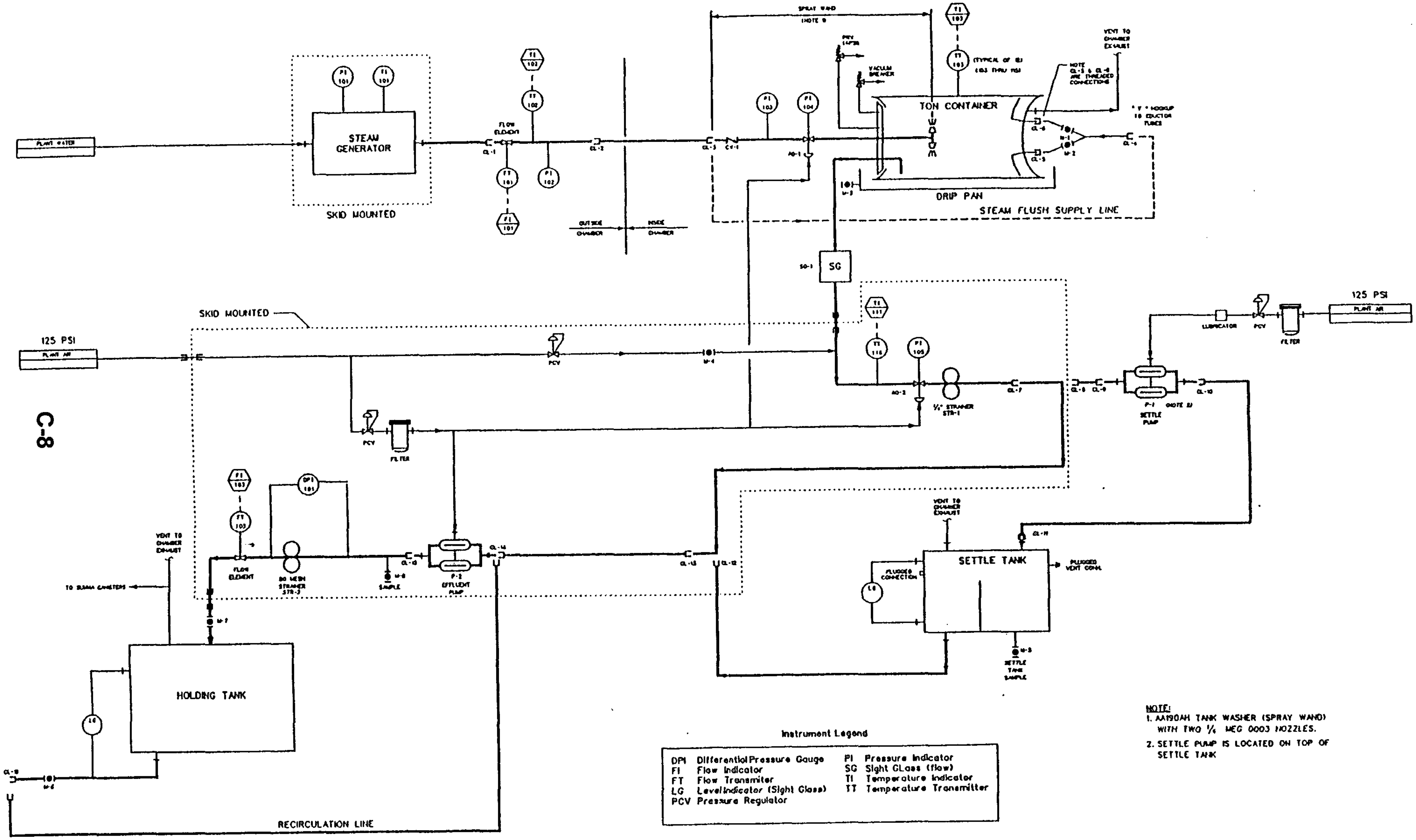
ENCLOSURE 1

As Built Drawing for the 2nd HD TC Cleanout Demonstration

HD TON CONTAINER CLEANOUT DEMONSTRATION



Instrument Legend

DPI	Differential Pressure Gauge	PI	Pressure Indicator
FI	Flow Indicator	SG	Sight GLass (flow)
FT	Flow Transmitter	TI	Temperature Indicator
LG	LevelIndicator (Sight Glass)	TT	Temperature Transmitter
PCV	Pressure Regulator		

NOTE:
1. AA190AH TANK WASHER (SPRAY WAND) WITH TWO 1/4 MEG 0003 NOZZLES.
2. SETTLE PUMP IS LOCATED ON TOP OF SETTLE TANK

Figure 1-2 As-Built Drawing

ENCLOSURE 2

Demonstration Procedures for the 2nd HD TC Cleanout Demonstration

DEMONSTRATION PROCEDURES FOR THE 2nd HD TCC

2.5 Demonstration Procedures

Follow the SOP entitled "HD TC Cleanout Demonstration" and use the data collection sheets in Appendix D to proceed with the demonstration. Perform the following steps.

PHASE I: REMOVAL OF SOLIDS

Step A: Initial Characterization

1. Inspect the TC interior with the hand held video camera and take photographs. Record observations.

2. Through the 10 inch hole cut into the TC, use a scoop to obtain the necessary solid samples as stated in the analytical plan (Paragraph 2.8) and the data pages (Appendix D).

3. Position the TC such that the side indicated as the "CASY bottom" is located 90 degrees (3:00 position) and the eductor tubes are horizontal.

4. Clamp the interface plate to the TC. Insert the spray lance and nozzle assembly into the TC. Position the nozzle at the 3/4 position and set the angular position of the wand to the initial angular position (angular position 0).

5. Install the settle tank bypass line (if not already installed) and open/close the required valves to ensure the settle tank is bypassed.

Step B: Eductor Tube Flush to Remove Heel in Tubes

6. Remove the valves and set aside.

7. Install the "Y hookup" lines to the two eductor tubes. Ensure that the TC vent line is open.

8. Set the steam generator to steam.

9. Close the valve on one of the "Y hookup lines" to the eductor and flush the other eductor tube with steam for 5 minutes. Re-open the valve after flushing.

10. Flush the other eductor tube by closing the valve on the other line (to the eductor that was already flushed). Flush steam through the second eductor tube for 5 minutes.

11. Stop the flow of steam.

12. Remove the "Y hookup" and install 2 pipe plugs where the valves were connected.

Step C: Hot Water Spray to Remove Heel (1st 30 minutes)

13. Close the required valves to ensure filling of the TC. Ensure that the vent line on the TC is open.

14. (Time = 0 min) Set the hot water generator for 5 gpm and 90°C (+/-5°C) 194°F at the maximum pressure of the nozzles (up to 3000 psi). Record the actual performance characteristics of the hot water generator (pressure, temperature, flowrate) as identified in the Data Sheets.

15. Allow the TC to fill up for 10 min.

16. (Time = 9 min) Open the required valves on the effluent. Turn the effluent pump on. Prime the pump if needed.

17. Open the valves to the SUMMA canisters to begin sampling on the 100 ml/min (60 min sample) SUMMA and the 34 ml/min (180 min) SUMMA. Allow the SUMMA canisters to continue sampling for the required duration once the effluent is collected in the hold tank.

18. (Time = 10 min) Purge approximately 250 ml into a bucket or jar to be emptied into holding tank at a convenient time. Obtain a 200 ml liquid sample through the sample port. Determine the pH of the effluent liquid sample when it is collected. Collect solids from the coarse and fine strainers (switch the strainer flow to the second basket, remove the first basket and place in a bag and steel container, and install a clean basket). Weigh the removed strainer baskets after this stage of cleaning is completed. If necessary, remove the solids from the strainer baskets and place in a sample jar.

19. At 10 minute intervals, record the necessary data (temperatures, pressures, flowrates) as identified by the Data Sheets.

20. (Time = 15 min) Move the nozzle to the 1/4 position.

21. (Time = 20 min) Obtain liquid and solid samples as stated in Step 18.

23. (Time = 30 min) Obtain liquid and solid samples as stated in Step 18.

24. Stop hot water spraying.

25. Disconnect vent line and install plug into the threaded hole.

26. Set up system for hold tank recirculation. Recirculate contents for 30 min and obtain a one liter sample in addition to a 250 ml sample. Also, collect a 5 gallon sample in a 5 gallon plastic container (this sample will be stored at the CTF).

Step D: Characterization and DAAMS (if necessary)

27. Inspect the TC interior with the hand held video camera and take photographs. Record observations.

28. Monitor the TC level of vapor contamination with DAAMS tubes if the interior is visibly clean. The DAAMS requirement may be waived if the chamber monitoring indicates high agent readings. MINICAMS may be used to indicate the approximate agent level in the TC. If MINICAMS readings are below 1 TWA, then DAAMS testing can be done. DAAMS testing requires the following:

    a. Allow the TC to cool and dry.
    b. Install the interface plate and setup for the DAAMS tubes, and close vent line valve to TC.
    c. Allow the TC to be closed for at least 4 hours above 70°F.
    d. Install the DAAMS tubes and allow 2 hours for sampling.

PHASE II: DECONTAMINATE TC TO 3X

Step E: Hot Water Spray to Achieve 3X (2nd 30 minutes)

29. Install the wand and nozzle assembly and keep nozzle at the current position (3/4 or 1/4 position).

30. Remove the settle tank bypass line and open/close the required valves to ensure the effluent flows through the settle tank.

31. (Time = 0 min) Set the hot water generator for 5 gpm and 194°F (90°C) at the maximum pressure of the nozzles (up to 3000 psi). Record the actual performance characteristics of the generator (pressure, temperature, flowrate). At 15 minute intervals, record the necessary data (temperatures, pressures, flowrates) as identified by the Data Sheets.

32. Turn the effluent and settle pumps on.

33. (Time = 15 min) Obtain liquid samples as stated in Step 18.

34. Move the nozzle to the other position (3/4 or 1/4 position).

35. (Time = 30 min) Obtain solid samples from strainers as stated in Step 18, and obtain a 250 ml sample from the settle tank after a total of 60 minutes hot water spraying.

36. Shut down the hot water generators.

37. Remove the wand and nozzle assembly and the interface plate.

Step F: Characterization and DAAMS

38. Inspect the TC interior with the hand held video camera and take photographs. Record observations.

39. Monitor the TC level of vapor contamination with DAAMS tubes if the interior is visibly clean. The DAAMS requirement may be waived if the chamber monitoring indicates high agent readings. MINICAMS may be used to indicate the approximate agent level in the TC. If MINICAMS readings are below 1 TWA, then DAAMS testing can be done. DAAMS testing requires the following:

    a. Allow the TC to cool and dry.
    b. Install the interface plate and setup for the DAAMS tubes.
    c. Allow the TC to be closed for at least 4 hours above 70°F.
    d. Install the DAAMS tubes and allow 2 hours for sampling.

Step G: Hot Water Spray to Achieve 3X (Additional 30 minutes)

40. Repeat Phase II (steps E & F) until the DAAMS analysis results indicate that the interior of the TC is in a 3X condition.

41. Obtain a 250 ml settle tank sample after TC has been determined to be in a 3X condition.

42. Set up system for hold tank recirculation. Recirculate the contents of the holding tank for 30 minutes and obtain a 250 ml sample.

43. Inspect the 3X TC interior with the hand held video camera and take photographs. Record observations.

44. Weigh the 3X TC.

Samples will be taken at the following locations and times:

Event	Elapsed Time for Step (min)	Total Time (min)	Notes
Characterization			
Sample 1	-	-	Tap Water Benchmark
Sample 2	-	-	Benchmark SUMMA Canister
Sample 3	-	-	Initial SUMMA Canister
Sample 4	-	-	Scoop (top of peak, for analysis)
Sample 5	-	-	Scoop (close to wall, for analysis
Sample 6	-	-	Scoop (for analysis)
Sample 7	-	-	Scoop (top of peak, for backup)
Sample 8	-	-	Scoop (close to wall,for backup)
Sample 9	-	-	Scoop (for backup)
Eductor Tube Flush			
(No samples during this stage)	-	10	-
1st 30 min of Hot Water Spraying			
Sample 10	10	20	Liquid
Sample 11	10	20	Coarse Strainer Solid
Sample 12	10	20	Fine Strainer Solid
Sample 13	20	30	Liquid
Sample 14	20	30	Coarse Strainer Solid
Sample 15	20	30	Fine Strainer Solid
Sample 16	30	40	Liquid
Sample 17	30	40	Coarse Strainer Solid
Sample 18	30	40	Fine Strainer Solid
Sample 19	30	40	Hold Tank Sample (note: there will be one 250 ml sample, two 500 ml samples, one 5 gal sample & will be labelled 19A, 19B, 19C, 19D)
2nd 30 min Hot Water Spraying			
Sample 20	45	55	Liquid
Sample 21	60	70	60 min SUMMA*
Sample 22	60	70	Fine Strainer Solids (if any)
Sample 23	60	70	Settle Tank Sample
Sample 24	60	70	DAAMS (if necessary)

Event	Elapsed Time for Step (min)	Total Time (min)	Notes
Additional Hot Water Spraying			
Sample 25	75	85	Liquid
Sample 26	90	100	Fine Strainer Solids (if any)
Sample 27	90	100	DAAMS
Sample 28	105	115	Liquid
Sample 29	120	130	Fine Strainer Solids (if any)
Sample 30	120	130	DAAMS
Sample 31	135	145	Liquid
Sample 32	150	160	Fine Strainer Solids (if any)
Sample 33	150	160	DAAMS
Sample 34	165	175	Liquid
Sample 35	180	180	180 min SUMMA Canister*
Sample 36	180	190	Fine Strainer Solids (if any)
Sample 37	180	190	DAAMS (if necessary)
Sample 38	195	205	Liquid
Sample 39	210	220	Fine Strainer Solids (if any)
Sample 40	210	220	DAAMS
Sample 41	225	235	Liquid
Sample 42	240	250	Fine Strainer Solids (if any)
Sample 43	240	250	DAAMS
Sample 44	255	265	Liquid
Sample 45	270	280	Fine Strainer Solids (if any)
Sample 46	270	280	DAAMS
Sample 47	285	295	Liquid
Sample 48	300	310	Fine Strainer Solids (if any)
Sample 49	300	310	DAAMS
Sample 50	315	325	Liquid
Sample 51	330	340	Fine Strainer Solids (if any)
Sample 52	330	340	DAAMS

Event	Elapsed Time for Step(min)	Total Time (min)	Notes
Sample 53	345	355	Liquid
Sample 54	360	370	Fine Strainer Solids (if any)
Sample 55	360	370	DAAMS
Sample 56	-	(when TC is 3X)	Settle Tank Sample
Sample 57	-	(when TC is 3X)	Hold Tank Sample

End of Demonstration

**Note:* The 60 min and 180 min SUMMAs are started once effluent is pumped into the hold tank. A sample is collected continously for the required duration.

ENCLOSURE 3

Data Analysis Methods & Procedures for the 2nd HD TC Cleanout Demonstration

ANALYTICAL PLAN FOR 2nd HD TC CLEANOUT

Page 1/2

2.8 Data Analysis Methods and Procedures

The Analytical methods to be used on the liquid, solid, and vapor samples are addressed in the Ton Container Survey and Ton Container Cleanout Analytical Test Plan (PMAT&A, 1995).

The anlaytical methods to be used on the various samples are as follows:

Sample Type	Analysis	Lab & Analyst	Test Method	Notes
Tap Water	Metals/Mecury	ACT (Herd)	TC-05	1
Neat HD	Flash Point	PPT (Butrow)	EC-01	2
	Metals/Mercury	ACT (Herd)	TC-05	1
	Landban Compounds	ACT	HN-04	
	Purity	ACT (Vickers)	TC-03	
HD Characterization	HD Agent	ACT (Vickers)	TC-06	3
Solid Samples	Metals/Mercury	ACT (Herd)	TC-05	1
	NMR	ACT (Szafranic)	see note	4
Solid Effluents	HD Agent	ACT	TC-06	
(strainers)	Metals/Mercury	ACT (Herd)	TC-05	1
	NMR	ACT (Szafranic)	see note	4
	Particle Size Distribution	EAI or ACT, see note	ASTM C325-81	5
Liquid Effluents	Low Level HD Agent	ACT	HN-02	
	TDG	ACT (Ince)	HN-06	
	Organics	ACT	HN-04	
	Metals/Mercury	ACT (Herd)	TC-05	1
	NMR	ACT (Szafranic)	see note	4
	Total Suspended Solids	ACT	B-08	
Settle Tank Samples	Low Level HD Agent	ACT	HN-02	
& Hold Tank Samp.	TDG	ACT (Ince)	HN-06	
(note-settle tank will	Organics	ACT	HN-04	
be liquid & solid,	Metals/Mercury	ACT (Herd)	TC-05	
hold tank is liquid)	NMR	ACT (Szafranic)	see note	4
	Total Suspended Solids	ACT	B-08	
	Particle Size Distribution	EAI or ACT (see note)	ASTM C325-81	5

ANALYTICAL PLAN FOR 2nd HD TC CLEANOUT

Sample Type	*Analysis*	*Analyst*	*ATP Method*	*Notes*
SUMMA Canister	HD Agent	ACT (Pleva)	TC-02	
	VOC's	ACT (Pleva)	TC-02	
DAAMS or Resin Tubes	HD Agent	EAI (Gonzalez) or ACT (Pleva)?	see note	6

Notes:

1. A full scan Metals/Mercury analysis will be done on the tap water sample, the neat agent sample, one HD characterization solid sample (heel), one solid effluent sample (strainer), one liquid effluent, one settle tank sample, and one hold tank sample. Other samples will be analyzed for a partial scan of metals. This partial scan will include metals that were present in the full scan and metals that were present for a similar sample from the first HD TC Cleanout Demonstration.

2. Flash point analysis will be done on the HD ton container from the TC Survey that is identified as having the "worst case landbans".

3. Only two of the three HD characterization solid samples will be analyzed for content of agent. There will also be three solid samples taken for backup, if needed.

4. The Request for Analysis and Results Sheet (Form 49) will state exactly what is to be analyzed for NMR and Organics. For example, "determine known complex organic compounds, including the sulfonium ion, above 5%". Not all effluent samples will be analyzed for NMR due to the complexity and time necessary for this analysis; however the critical samples, such as the initial effluents, mid-way (to 3X) effluents, and tank samples, will be analyzed.

5. Particle Size Distribution will be determined using ASTM C325-81 and will be done by either the EAI Lab at E3726 or the ACT lab.

6. The vapor sample will be taken using either DAAMS or Tenax resin tubes. If the EAI Lab at E3726 is set up to analyze DAAMS tubes by the start of the test, then they will be utilized. If not, the ACT lab will analyze DAAMS tubes. If the ACT lab is not set up to analyze DAAMS tubes, Tenax resin tubes will be used in lieu of DAAMS tubes.

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ENCLOSURE 4

Data Sheets for the 2nd HD TC Cleanout Demonstration

APPENDIX D
HD Ton Container Cleanout Demonstration
Notebook No.: ________ Date: ____________
Subtest No.: 3

Subtest No. 3 Test Data Sheets

Purpose: Identify and record all information required for successful completion of the HD TC cleanout demonstration.

Preparation of an Empty TC

HD TC Information	
Stamped Tare Weight	lbs
Stamped TC Information	
Weight of Removed HD (from CTF)	lbs
Weight of Drained TC (from CTF)	lbs
Weight of Drained TC (at E3566)	lbs
Estimated Weight of HD Residue	lbs
Level of Contamination	
Cutout Weight	lbs
Observations	
Depth of Bottom Residue	in.
Thickness of Side/Bottom Scale	in.

Signature ______________________ Title ____________________ Date __________

Notebook No.: ________ Date: ____________

Subtest No.: 3

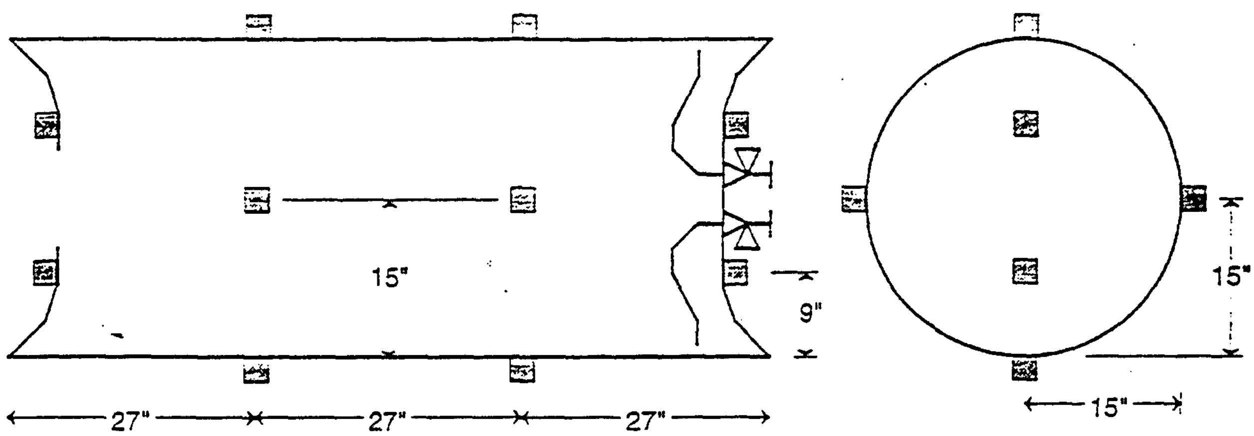
Characterization Samples				
Sample No.	Sample ID	Time	Weight (lbs)	Sample Observations
C-03-01-HH-____	Tap Water Benchmark	-		
C-03-02-HH-____	Benchmark SUMMA®	-		
C-03-03-HH-____	Initial SUMMA®	-		
C-03-04-HH-____	Scoop	-		Top of Peak
C-03-05-HH-____	Scoop	-		Bot of heel, close to wall
C-03-06-HH-____	Scoop	-		
C-03-07-HH-____	Scoop	-		Top of Peak
C-03-08-HH-____	Scoop	-		Bot of heel, close to wall
C-03-09-HH-____	Scoop	-		
C-03-F__-HH-____				
C-03-F__-HH-____				

Signature ______________________ Title ______________________ Date __________

APPENDIX D
HD Ton Container Cleanout Demonstration
Notebook No.: ________ Date: ____________
Subtest No.: 3

Thermocouple Locations

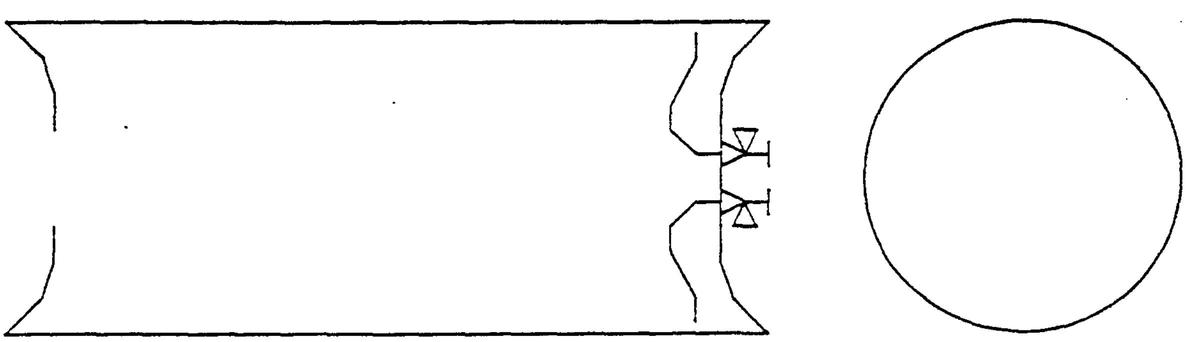
Planned



= Thermocouple

Side View End View

Actual



Side View End View

Signature ______________________ Title ______________________ Date __________

APPENDIX D
HD Ton Container Cleanout Demonstration

Notebook No.: ________ Date: ____________

Subtest No.: 3

Eductor Tube Flushing

Preparation for Eductor Tube Flushing	
Weight of Removed Valves	lbs.

Hot Water/Steam Generator			
Parameter	Set	Actual	Date & Time Recorded
Temperature	300°F (149°C)	°F	
Pressure	2500 psig	psig	
Flowrate	1 gpm	gpm	

Eductor Tube Flushing Observations							
Period	Steaming Time (min)	Date & Time	Observ. Time (min)	Hot Water or Steam Temp (F)	Flow rate (gpm)	Avg Surface Temp (F)	Effluent Temp (F)
1 1st Tube	5				1 gpm		
2 2nd Tube	10				1 gpm		

Signature ______________________ Title ____________________ Date __________

APPENDIX D
HD Ton Container Cleanout Demonstration

Notebook No.: ________ Date: ____________

Subtest No.: 3

Pressurized Hot Water Spraying

Hot Water Generator		
Parameter	Set	Actual
Flowrate	5 gpm	gpm
Pressure	3000 psi	psi
Temperature	194°F (90°C)	°F

Hot Water Generator						
Period	Spraying Time (min)	Date & Time (start/stop)	Actual Time (min)	Flow Rate (gpm)	Pressure (psi)	Temp (°F)
3 (10)	10					
4 (20)	20					
5 (30)	30					
6 (15)	45					
7 (30)	60					
8 (15)	75					
9 (30)	90					
10 (15)	105					
11 (30)	120					
12 (15)	135					
13 (30)	150					
14 (15)	165					
15 (30)	180					

Signature ______________________ Title ____________________ Date ________

APPENDIX D
HD Ton Container Cleanout Demonstration

Notebook No.: ________ Date: ____________

Subtest No.: 3

Hot Water Generator (Continued)						
Period	Spraying Time (min)	Date & Time (start & stop)	Actual Time (min)	Flow Rate (gpm)	Pressure (psi)	Temp (°F)
16 (15)	195					
17 (30)	210					
18 (15)	225					
19 (30)	240					
20 (15)	255					
21 (30)	270					
22 (15)	285					
23 (30)	300					
24 (15)	315					
25 (30)	330					
26 (15)	345					
27 (30)	360					

Signature ______________________ Title ____________________ Date __________

APPENDIX D
HD Ton Container Cleanout Demonstration

Notebook No.: ________ Date:____________

Subtest No.: 3

Pressurized Hot Water Cleaning Observations								
Period	Total Spray Time (min)	Observ Time (min)	Avg Surface Temp (°F)	Effluent Temp (°F)	Fine Strainer Diff Press (psi)	Avg Effluent Flow (gpm)	Settle Tank Level (gal)	Hold Tank Level (gal.)
3 (10)	10							
4 (20)	20							
5 (30)	30							
6 (15)	45							
7 (30)	60							
8 (15)	75							
9 (30)	90							
10 (15)	105							
11 (30)	120							
12 (15)	135							
13 (30)	150							
14 (15)	165							
15 (30)	180							
16 (15)	195							
17 (30)	210							
18 (15)	225							
19 (30)	240							
20 (15)	255							
21(30)	270							
22(15)	285							
23 (30)	300							
24 (15)	315							
25 (30)	330							
26 (15)	345							
27(30)	360							

Signature ____________________ Title ____________________ Date __________

APPENDIX D
HD Ton Container Cleanout Demonstration

Notebook No.: ________ **Date:** ____________

Subtest No.: 3

Pressurized Hot Water Cleaning Samples						
Sample No.	Sample ID	Clock Time	Spray Time (min)	Volume (mL)	Weight (lbs)	Sample pH and Observations
C-03-10-HH-____	Liquid		10			
C-03-11-HH-____	Coarse Strainer		10			
C-03-12-HH-____	Fine Strainer		10			
C-03-13-HH-____	Liquid		20			
C-03-14-HH-____	Coarse Strainer		20			
C-03-15-HH-____	Fine Strainer		20			
C-03-16-HH-____	Liquid		30			
C-03-17-HH-____	Coarse Strainer		30			
C-03-18-HH-____	Fine Strainer		30			
C-03-19-HH-____	Hold Tank		30			Note: take 4 samples (ABCD)
C-03-20-HH-____	Liquid		45			
C-03-21-HH-____	60 min SUMMA		60			
C-03-22-HH-____	Fine Strainer		60			
C-03-23-HH-____	Settle Tank		60			
C-03-24-HH-____	DAAMS		60			
C-03-25-HH-____	Liquid		75			
C-03-26-HH-____	Fine Strainer		90			
C-03-27-HH-____	DAAMS		90			

Signature ______________________ Title ____________________ Date __________

APPENDIX D
HD Ton Container Cleanout Demonstration

Notebook No.: ________ Date: ____________

Subtest No.: 3

Pressurized Hot Water Cleaning Samples (Continued)						
Sample No.	Sample ID	Clock Time	Spray Time	Volume (mL)	Weight (lbs)	Sample pH and Observations
C-03-28-HH-____	Liquid		105			
C-03-29-HH-____	Fine Strainer		120			
C-03-30-HH-____	DAAMS		120			
C-03-31-HH-____	Liquid		135			
C-03-32-HH-____	Fine Strainer		150			
C-03-33-HH-____	DAAMS		150			
C-03-34-HH-____	Liquid		165			
C-03-35-HH-____	180 min SUMMA		180			
C-03-36-HH-____	Fine Strainer		180			
C-03-37-HH-____	DAAMS		180			
C-03-38-HH-____	Liquid		195			
C-03-39-HH-____	Fine Strainer		210			
C-03-40-HH-____	DAAMS		210			
C-03-41-HH-____	Liquid		225			
C-03-42-HH-____	Fine Strainer		240			
C-03-43-HH-____	DAAMS		240			
C-03-44-HH-____	Liquid		255			
C-03-45-HH-____	Fine Strainer		270			
C-03-46-HH-____	DAAMS		270			

Signature ______________________________ Title _________________________ Date __________

APPENDIX D
HD Ton Container Cleanout Demonstration

Notebook No.: ________ **Date:** ____________

Subtest No.: 3

Pressurized Hot Water Cleaning Samples (Continued)						
Sample No.	Sample ID	Clock Time	Spray Time	Volume (mL)	Weight (lbs)	Sample pH and Observations
C-03-47-HH-____	Liquid		285			
C-03-48-HH-____	Fine Strainer		300			
C-03-49-HH-____	DAAMS		300			
C-03-50-HH-____	Liquid		315			
C-03-51-HH-____	Fine Strainer		330			
C-03-52-HH-____	DAAMS		330			
C-03-53-HH-____	Liquid		345			
C-03-54-HH-____	Fine Strainer		360			
C-03-55-HH-____	DAAMS		360			
C-03-56-HH-____	Settle Tank		-			
C-03-57-HH-____	Hold Tank		-			
C-03-F__-HH-____						
C-03-F__-HH-____						
C-03-F__-HH-____						

Signature ______________________ Title ______________________ Date __________

Notebook No.: ________ **Date:** ____________
Subtest No.: 3

Other Pressurized Hot Water Cleaning Information	
Duration of Cleaning Operation (actual)	
Amount of Liquid Effluents Generated	gal.
Amount of Solids Collected	lbs
Level of Vapor Contamination @ 60 min	
Level of Vapor Contamination @ 90 min	
Level of Vapor Contamination @ 120 min	
Level of Vapor Contamination @ 150 min	
Level of Vapor Contamination @ 180 min	
Level of Vapor Contamination @ 210 min	
Level of Vapor Contamination @ 240 min	
Level of Vapor Contamination @ 270 min	
Level of Vapor Contamination @ 300 min	
Level of Vapor Contamination @ 330 min	
Level of Vapor Contamination @ 360 min	

Pressurized Hot Water Cleaning Gas Sampling Information		
	Set	Actual
SUMMA® (60 minute sample)	100 mL/min	mL/min
SUMMA® (180 minute sample)	34 mL/min	mL/min

Signature ______________________ Title ______________________ Date __________

APPENDIX D
HD Ton Container Cleanout Demonstration

Notebook No.: ________ Date: ____________
Subtest No.: 3

Inspection of the Cleaned TC

HD TC Information	
Weight of Cleaned/Steamed TC	lbs
Weight of Residue Removed (= Drained Weight - Cutout Weight - Valve Weight - Cleaned/Steamed Weight)	lbs
Level of Vapor Contamination	

Signature ______________________ Title ______________________ Date __________

APPENDIX D
HD Ton Container Cleanout Demonstration

Notebook No.: ________ **Date:** ____________

Subtest No.: 3

System Observations: (Notes about the physical operation of the demonstration)

Signature ____________________________ Title ________________________ Date __________

APPENDIX D
HD Ton Container Cleanout Demonstration

Notebook No.: ________ Date: ____________

Subtest No.: 3

Deviations from the Demonstration Plan

Signature ______________________ Title ____________________ Date ________

APPENDIX D

HD Ton Container Cleanout Demonstration

Notebook No.: ________ Date: ____________

Subtest No.: 3

Attach disk(s) containing:

Surface and effluent temperature versus time

Differential pressure versus time

Disk Title: ______________________

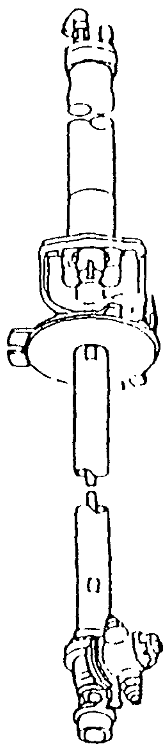
This data will not be reproduced except in full, without written permission of the laboratory.

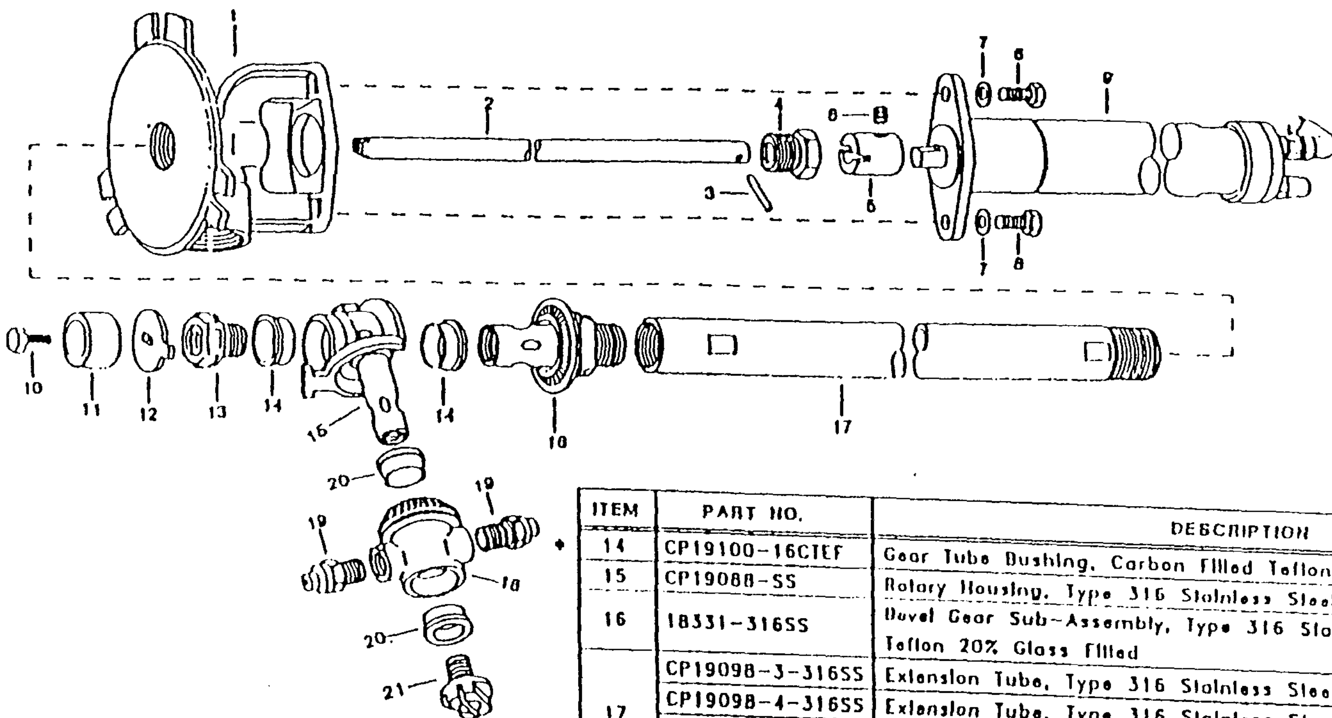
Client: PMAT&A
Address: Aberdeen Proving Ground, MD 21010-5423

Laboratory: ERDEC
Address: SCBRD-ENM-S E3706 Aberdeen Proving Ground, MD 21010-5423

Signature ____________________________ Title ________________________ Date __________

APPENDIX D
DRAWINGS



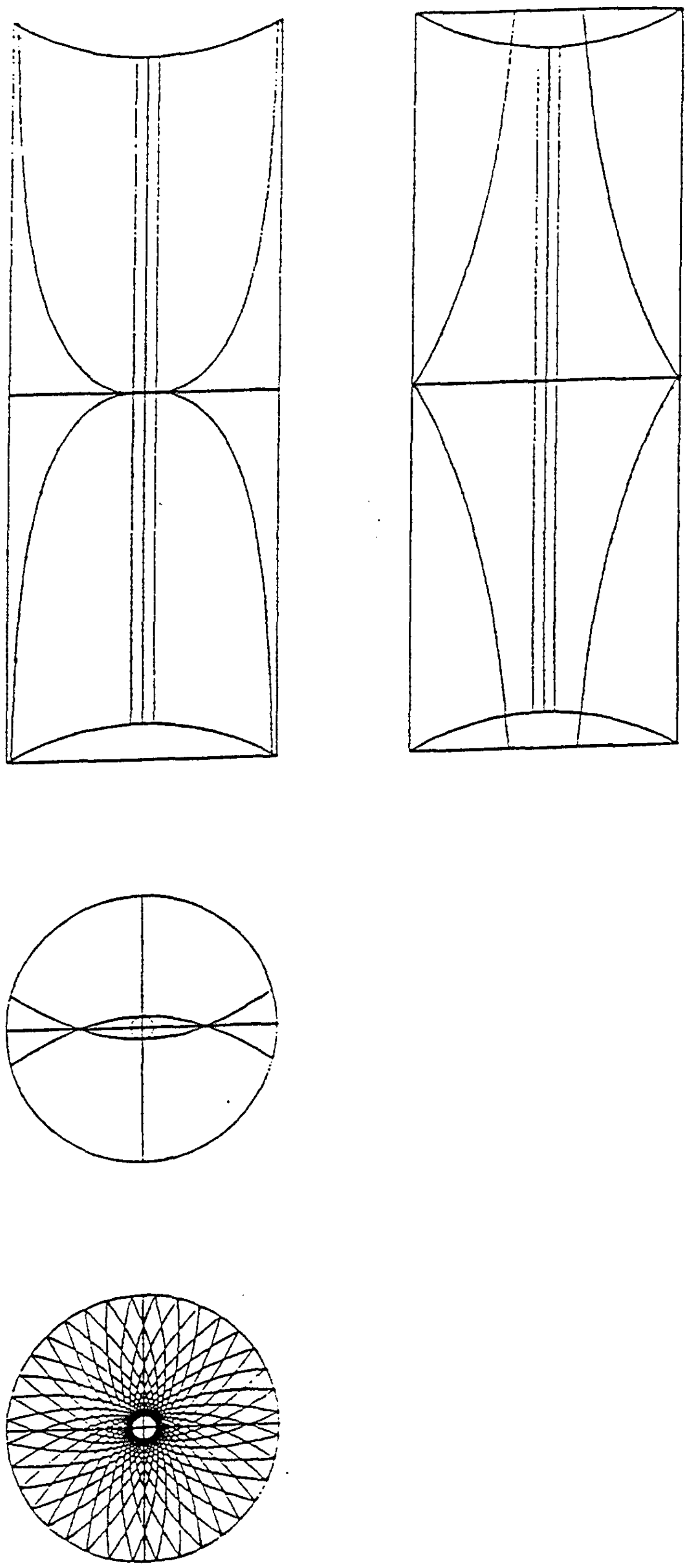


...O.	DESCRIPTION
...S	Liquid Inlet Body-Mounting Plate Sub-Assembly, Type 316 Stainless Steel (1" NPT Conn.)
...-316SS	Shaft, Type 316 Stainless Steel (For Model #AA190AH-3)
...-316SS	Shaft, Type 316 Stainless Steel (For Model #AA190AH-4)
...-316SS	Shaft, Type 316 Stainless Steel (For Model #AA190AH-6)
...-316SS	Shaft, Type 316 Stainless Steel (For Model #AA190AH-8)
...S	Groove Pin, Stainless Steel
...SS	Upper Bushing Retainer Sub-Assembly, Type 316 Stainless Steel and Carbon Filled Teflon
...16SS	Coupling, Type 316 Stainless Steel

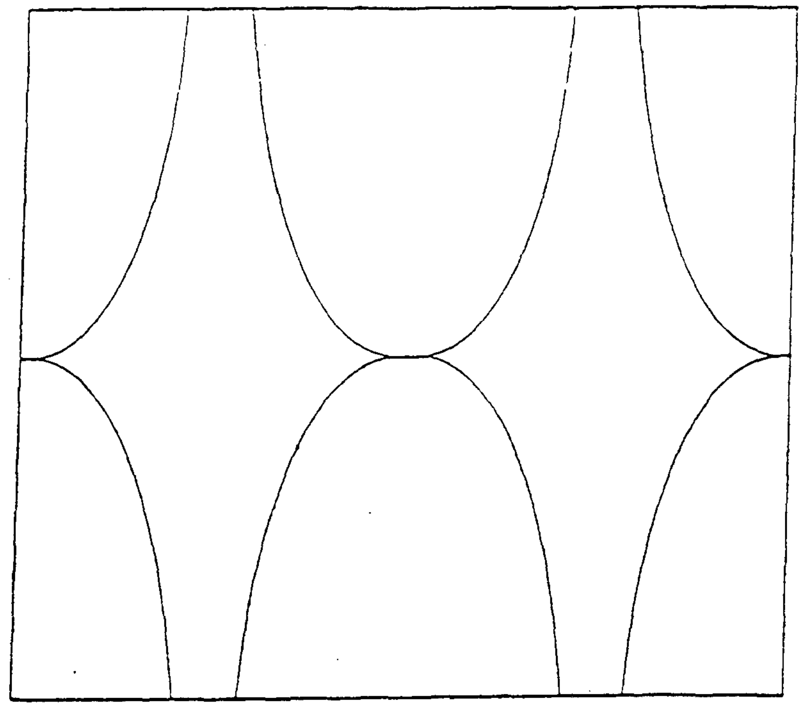
ITEM	PART NO.	DESCRIPTION
14	CP19100-16CTEF	Gear Tube Bushing, Carbon Filled Teflon (2 Req'd)
15	CP19088-SS	Rotary Housing, Type 316 Stainless Steel
16	18331-316SS	Bevel Gear Sub-Assembly, Type 316 Stainless Steel a... Teflon 20% Glass Filled
17	CP19098-3-316SS	Extension Tube, Type 316 Stainless Steel (For Model ...
	CP19098-4-316SS	Extension Tube, Type 316 Stainless Steel (For Model ...
	CP19098-6-316SS	Extension Tube, Type 316 Stainless Steel (For Model ...
	CP19098-8-316SS	Extension Tube, Type 316 Stainless Steel (For Model ...
18	CP19086-SS	Nozzle Hub, Type 316 Stainless Steel (N.P.T. Conn.)
19	**	Spray Nozzle, Solid Stream WashJet Nozzle (2 Req'd), Stainless Steel (1/4" NPT Conn.) See Ind. Catalog and D.S. #15659 (WashJet)
20	CP19104-16CTEF	Nozzle Hub Bushing, Carbon Filled Teflon (2 Req'd)
21	CP19105-316SS	Rotary Housing Plug, Type 316 Stainless Steel (Left H...

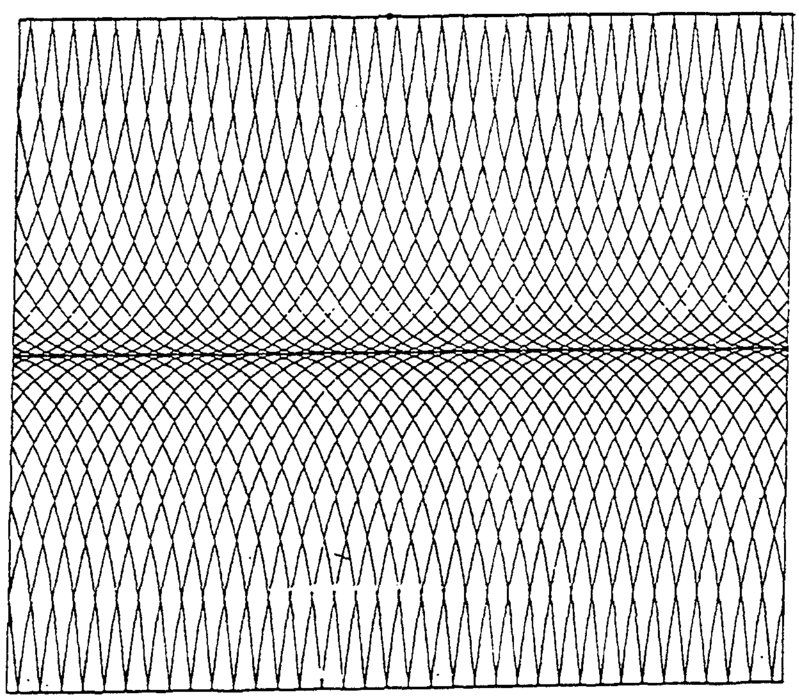
No. AA190AH-3+**, Motorized Rotary Tank Washer (3' Extension)
No. AA190AH-4+**, Motorized Rotary Tank Washer (4' Extension)
No. AA190AH-6+**, Motorized Rotary Tank Washer (6' Extension)
No. AA190AH-8+**, Motorized Rotary Tank Washer (8' Extension)

Spray Patterns Created by the Spray Lance and Nozzle Assembly

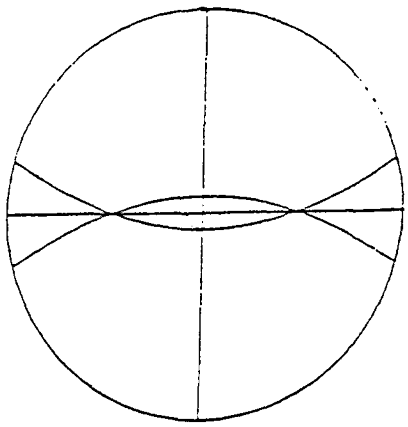


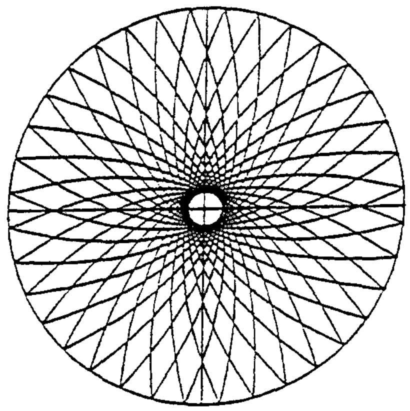
Spray Patterns Created by the Spray Lance and Nozzle Assembly

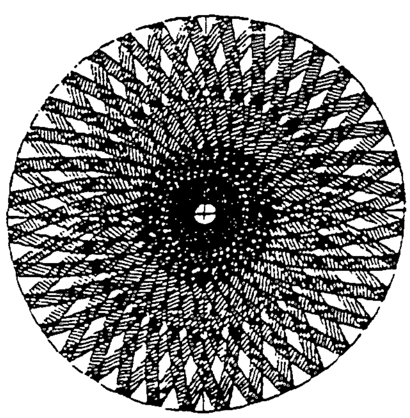




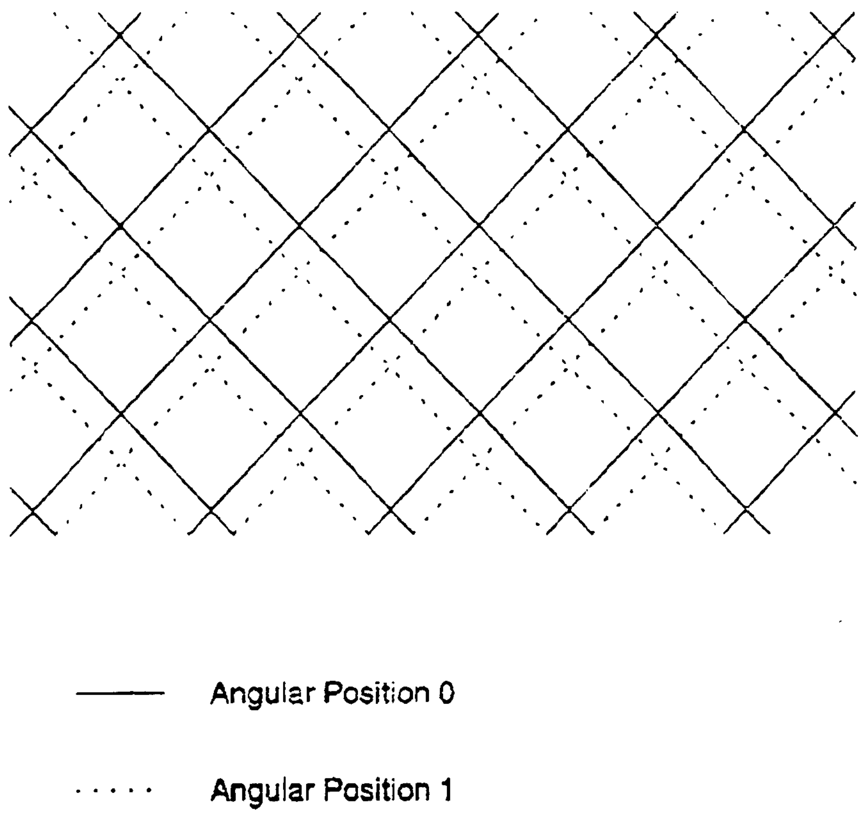
Spray Patterns Created by the Spray Lance and Nozzle Assembly



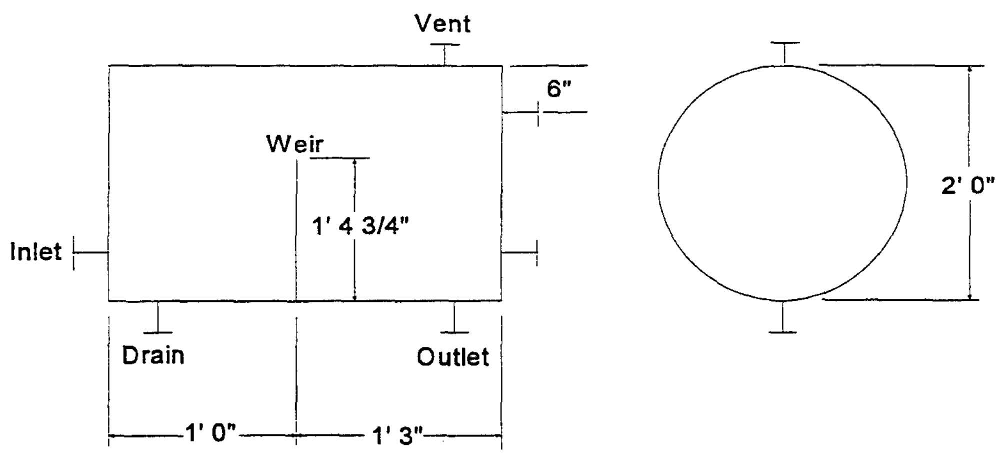




Crosshatches Produced at Angular Positions 0 and 1



Settle Tank



MATERIAL: 1/8" 316L Stainless Steel

APPENDIX E
CALIBRATION AND VERIFICATION RECORDS

Copies of all calibration and verification records can be found in the Ton Container Decontamination and Disposal Program Demonstration Report: A Second Mustard Agent Ton Container (TC Serial #D94102) - Volume 2 (Reference 12).

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APPENDIX F
DATA COLLECTED

All original data can be found at the ERDEC Toxic Test Chamber in building E3566 in the E3566 Log Book #D1 and #D2: Ton Container Cleanout Demonstrations (References 9 and 10), and in the E3566 "Second HD Ton Container Cleanout Demonstration Binder" (Reference 11), which contains all original data sheets, plots, and analytical results.

Copies of all data and analytical results can be found in the Ton Container Decontamination and Disposal Program Demonstration Report: A Second Mustard Agent Ton Container (TC Serial #D94102) - Volume 2 (Reference 12).

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APPENDIX G
DEMONSTRATION DEVIATIONS

The problems encountered in the second HD TC Cleanout Demonstration, which may have affected the results of the demonstration, are discussed in detail in Section 3 and are briefly summarized below.

- During the first 30 minutes of hot water spraying, a hose to the hand-held wand of the Hydroblaster hot water generator failed, causing low flow, low pressure water to be introduced to the TC for approximately 3 minutes. While fixing the problem, approximately 27 gallons of water remained in the TC for 3 hours and 18 minutes, which may have affected the results of the demonstration.

- The two SUMMA® canisters which were to collect the vapor sample off the holding tank, did not function correctly and therefore, a sample was not collected at the proper time. Instead, a sample was taken before the actual cleaning of the TC; therefore, no sample was obtained from the hold tank once effluent was collected.

- After 30 minutes of hot water spraying, the hot water generators were not properly shut down, thus causing approximately 10 gallons of water to enter the TC. This water remained in the TC for a number of days (since the effluent pump had also failed), which may have affected the results of the demonstration.

- The effluent pump failed during the recirculation of the hold tank after 30 minutes of hot water spraying, possibly causing inadequate mixing of the hold tank contents. Therefore, a "representative" sample of the hold tank may not have been obtained.

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